ENCYCLOPEDIA
OF
CHEMICAL REACTIONS

COMPILED AND EDITED BY

C. A. JACOBSON

Professor of Chemistry Emeritus
West Virginia University

VOLUME III

COBALT
COLUMBIUM
COPPER
DIDYMIUM
DYSPROSIUM
ERBIUM
EUROPIUM
FLUORINE
GADOLINIUM
GALLIUM
GERMANIUM
GOLD
HAFNIUM
HOLMIUM
HYDROGEN
ILLINIUM
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IODINE
IRIDIUM

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Introduction

This is the third volume of a series intended ultimately to embrace the published reactions of the active chemical elements and their simpler compounds. Comparatively few chemical reactions will be found in the literature under the rarer elements, but whenever reactions of these elements are encountered in standard chemical journals they will be included in this compilation.

The reactions under aluminum, antimony, arsenic, barium, beryllium, bismuth, boron and bromine were included in Volume I, and cadmium, calcium, carbon, cerium, cesium, chlorine and chromium were covered in Vol. II.

Since the "Encyclopedia of Chemical Reactions" is planned on the alphabetical system throughout, the following elements have been included in Vol. III: cobalt, columbium, copper, didymium, dysprosium, erbium, europium, fluorine, gadolinium, gallium, germanium, gold, hafnium, holmium, hydrogen, illinium, indium, iodine and iridium. This volume contains 822 reactions under cobalt alone and 610 under copper; whereas holmium and illinium are represented by one reaction each and hafnium by two.

The introduction to Vol. II contains a discussion of coordinate valence reactions of cobalt and chromium, together with the abbreviations used. To save space, many of the complex reactants herein described have been written in abbreviated forms.

Whenever an element is known by two names, *e.g.*, columbium and niobium, the more common form has been used to the exclusion of the other.

Frequently equations are encountered in the literature that are unbalanced. Whenever it is a question of adding or substracting water to make the equation balance, this has been done, but in all these cases the equations so modified have been placed in parentheses. Frequently authors' names appear without initials, but this is not an error of the abstractors or editor; such references are incomplete in the original. Dates too are sometimes found to be wrong. In many cases where an author describes a product obtained he neglects to mention the accompanying by-products, although they are evident. In such cases the abstractor supplies the missing self-evident products and puts them in parentheses. This is why so many reactions are enclosed in parentheses.

In the process of assembling these reactions it was necessary to select abstractors who not only were enthusiastic about the completion of the work, but were willing to contribute of their time to abstract suitable reaction material found in the literature.

In order that the abstractors' work should not overlap that of others, it was necessary to assign to each person a certain journal or section of a journal to be covered. It often happened that an abstractor selected for this work, and willing to contribute his time to the undertaking, had no suitable journal from which to abstract. Half a dozen or more abstractors have been supplied with chemical journals from the Library of the West Virginia University. In this way it was possible to make available for abstracting purposes several rare journals for which no abstractor could be found in Morgantown.

In one instance over four hundred volumes of Liebig's *Annalen der Chimie* were loaned in small lots to one abstractor alone, who not only translated into English this huge mass of chemical German, of a very technical nature, but typed the translated material and sent it to the General Office of the E.C.R. for filing. Several other abstractors have done nearly as well, and it is with singular admiration that the Editor-in-Chief points out the unselfish and public-spirited nature of these men and women, who have contributed solely for the advancement of science, and without any thought of reward for themselves.

The "Encyclopedia" is not designed to present the latest views of chemistry and chemical technology, but rather to summarize the facts presented in the published literature. Its users will therefore have the opportunity to evaluate previous experimental work, when applied to present problems.

The editors regret to say that in spite of every attempt to eliminate duplicate entries in the body of the reaction material they have not been wholly successful in this respect. It was when such duplicates or near-duplicates were brought into juxtaposition that they noticed the similarity. However, these imperfections do not greatly impair the value of the work.

Since Vol. III contains a considerable number of coordinate reactions with cobalt it was considered advisable to include in the introduction a list of coordinate adjectives with their abbreviations as used in these reactions. These adjectives all end in o when occurring in the coordinate sphere, represented by parentheses. They are as follows:

Aceto [$C_2H_3O_2$]
Amino [NH_2]
Ammino [NH_3]
Aquo [H_2O]

Anilino [$C_6H_5NH_2$]
Benzylamino [C_7H_9N]
Bromo [Br]
Carbonato [CO_3]
Chloro [Cl]
Chromato [CrO_4]
Diethylenediamino [$(C_2H_8N_2)_2$] Also written [en_2]
Ethylenediamino [$C_2H_8N_2$] Also written [en]
Hydroxo [OH]
Imino [NH]
Isothiocyanato [NCS]
Malonato [$C_3H_2O_4$]
Nitrato [NO_3]
Nitrito [NO_2]
Ol [OH]
Oxalato [C_2O_4]
Peroxo [OCO]
Pyridino [C_5H_5N] Also written [py]
Sulfato [SO_4]
Sulfito [SO_3]

C. A. Jacobson.

MORGANTOWN, W. Va.
June 3, 1949

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Abbreviations

Ac	stands	for	acetate
$\bar{c}$	"	"	with
conc.	"	"	concentrated
Δ	"	"	heat
decomp.	"	"	decomposition
dil.	"	"	dilute
ϵ	"	"	electric current or spark
Et	"	"	ethyl
m–	"	"	meta
M	"	"	univalent metal
Me	"	"	methyl
N	"	"	normal
o–	"	"	ortho
p–	"	"	para
P	"	"	pressure
Ph	"	"	phenyl
p.p.m.	"	"	parts per million
sol.	"	"	solution
en	"	"	ethylenediamino
pn	"	"	propylenediamino
py	"	"	pyridino

COBALT

Co

B **III-1**

Cobalt is heated with boron in a porcelain tube to 1100–1200° in a stream of hydrogen.

(a) $Co + B \rightarrow CoB$

(b) $2Co + B \rightarrow Co_2B$

(c) $Co + 2B \rightarrow CoB_2$

B. DuJassonneix, Comp. rend., **145**, 240 (1907) *25*

Co

Br_2 **III-2**

Cobalt, heated to faint redness, reacts with bromine vapor yielding a green product.

$$Co + Br_2 \rightarrow CoBr_2$$

Berthelot, Ann. chim. phys. (2), **44**, 391 (1830) *25*

Co

Br_2 **III-3**

When metallic cobalt is treated with bromine at ordinary temperature and in the presence of water, cobaltous bromide is formed. The reaction requires approximately one week to reach completion.

$$Co + Br_2 \rightarrow CoBr_2$$

Walter N. Hartley, J. Chem. Soc. (London), **27**, 501 (1874) *48*

Co

Cl_2 **III-4**

Powdered cobalt when first warmed, burns in chlorine.

$$Co + Cl_2 \rightarrow CoCl_2$$

H. Rose, Pogg. Ann., **24**, 157 (1832) *25*

Co

Cu_3P_2 **III-5**

A mixture composed of one part of cobalt filings and ten parts of cupric phosphide is heated five minutes in the electric furnace.

$$4Co + Cu_3P_2 \rightarrow 2Co_2P + (3Cu)$$

Maronneau, Compt. rend., **130**, 657 (1900) *25*

Co

HNO_3 **III-6**

Cobalt is heated with concentrated nitric acid.

$$Co + 4HNO_3 \rightarrow Co(NO_3)_2 + 2NO_2 + 2H_2O$$

Richards and Baxter, Z. anorg. Chem., **22**, 221 (1899) *25*

Co

H_2O **III-7**

Cobalt, heated to glowing, decomposes water vapor.

$$Co + H_2O \rightarrow CoO + H_2$$

Regnault, Ann. chim. phys., **62**, 351 (1836) *25*

Co

I_2 **III-8**

Powdered cobalt and iodine heated together in a glass tube react to form a gray-green product.

$$Co + I_2 \rightarrow CoI_2$$

O. L. Erdmann, J. f. prakt. Chem., **7**, 354 (1836) *25*

Co

I_2 **III-9**

When metallic cobalt is heated with iodine in the presence of water, the olive-green, di-hydrated cobaltous iodide is formed.

$$Co + I_2 + 2H_2O \rightarrow CoI_2 \cdot 2H_2O$$

Walter N. Hartley, J. Chem. Soc. (London), **27**, 502 (1874) *48*

Co

KCN **III-10**

Cobalt when titrated with cyanide in the presence of potassium iodide forms a compound in which the ratio of cobalt to cyanide is 1:5.

(a) $Co + 5KCN + 2H_2O \rightarrow K_3Co(CN)_5 + (2KOH + H_2)$

(b) $Co + 5KCN + 3H_2O \rightarrow K_3Co(CN)_5 \cdot H_2O + (2KOH + H_2)$

Glasstone and Speakman, Analyst, **55**, 93 (1930)
Ref., B. S. Evans, Analyst, **62**, 377 (1937) *40*

Co

P **III-11**

Cobalt is heated with phosphorus in the absence of air.

$$2Co + P \rightarrow Co_2P$$

Zhemchzuzhnii and Schepeler, Z. anorg. Chem., **64**, 245 (1909) *25*

Co

Si **III-12**

90 parts of cobalt are heated with 10 parts of silicon in a carbon crucible in the electric furnace.

$$2Co + Si \rightarrow Co_2Si$$

Moissan, Compt rend., **121**, 621 (1895) *25*

Co

Si **III-13**

Silicon and cobalt react in the electric Moissan furnace to give the following silicides:

$$SiCo_2,\ SiCo,\ Si_2Co$$

P. Lebeau, Z. Elektrochem., **9**, 642 (1903) *86*

Co

$SiCl_4$ **III-14**

Silicon tetrachloride is passed over finely pulverized cobalt at a high temperature, but not exceeding 1200°.

$$2Co + SiCl_4 \rightarrow Co_2Si + (2Cl_2)$$

Vigouroux, Compt. rend., **121**, 683 (1895) *25*

Co

Te **III-15**

Cobalt telluride is formed when cobalt and tellurium unite in an inert atmosphere.

$$Co + Te \rightarrow CoTe$$

Margottet, Thése de Paris (1879)
Fabre, Compt. rend., **105**, 277 (1887)
C. A. Tibbals, Jr., J. Am. Chem. Soc., **31**, 908 (1909) *25*

Co^{++}

Cl^- **III-16**
HCl

Solutions containing cobaltous ions give a blue-green color when treated with excess chloride ion, as in hydrochloric acid, due to the formation of a complex anion, as shown by the fact that the cobalt migrates toward the anode.

$$Co^{++} + 4Cl^- \rightarrow [CoCl_4]^{--}$$

Howell, Phil. Mag., *48*, 833 (1924); Ref., Proc. Roy. Soc., *142*, A, 587 (1933). *57*

Co^{++}

3,5-dimethylpyrazole **III-17**

Pour a 2% solution of 3,5-dimethylpyrazole into a previously nearly neutralized (NaOH) cobalt solution. It should be still faintly acid. Add 5 cc. of 0.5*N* NaOH whereupon all the cobalt precipitates.

$$Co^{++} + 2C_5H_8N_2 \rightarrow \underline{Co(C_5H_7N_2)_2}$$

O. Heim, Anal. Ed., **2**, 38 (1930) *114*

Co^{++}

KNO_2 **III-18**

Nickel and cobalt may be separated from one another by treatment with potassium nitrite. Potassium cobaltinitrite will precipitate, whereas nickel will stay in solution. Nitric oxide is evolved during the reaction.

$$(Co(NO_3)_2 + 5KNO_2 + 2HNO_2 \rightarrow \underline{K_3Co(NO_2)_6} + NO + H_2O + 2KNO_3)$$

Richard Apjohn, J. Chem. Soc. (London), **27**, 105 (1874) *48*

$CoBr_2$

H_2 **III-19**

Cobaltous bromide is reduced by hydrogen when heated.

$$CoBr_2 + H_2 \rightarrow Co + (2HBr)$$

Carli, Atti. ac. pont Lincei., **33:2**, 94 (1924) *25*

$CoBr_2$

NH_3 **III-20**

An aqueous solution of cobalt (II) bromide added to excess ammonium hydroxide gives a bright red precipitate of cobalt dibromide hexammoniate.

$$CoBr_2 + 6NH_3 \rightarrow \underline{CoBr_2 \cdot 6NH_3}$$

Rammelsberg, Pogg. Ann., **55**, 245 (1842)
A. Werner and P. Karrer, Helv. Chim., **1**, 62 (1918) *25*

$CoBr_2 \cdot 6H_2O$

$AgNO_3$ **III-21**

Treatment of hexa-hydrated cobaltous bromide with silver nitrate results in the formation of cobaltous nitrate and silver bromide.

$$CoBr_2 \cdot 6H_2O + 2AgNO_3 \rightarrow Co(NO_3)_2 + \underline{2AgBr} + 6H_2O$$

Walter N. Hartley, J. Chem. Soc. (London), **27**, 501 (1874) *48*

$CoBr_2 \cdot 6H_2O$

KOH **III-22**

O_2

When hexa-hydrated cobaltous bromide is heated with potassium hydroxide in presence of air, the cobaltous salt is oxidized to cobaltic hydroxide by the oxygen of the air.

$$4CoBr_2 \cdot 6H_2O + 8KOH + O_2 \rightarrow 4Co(OH)_3 + 8KBr + 22H_2O$$

Walter N. Hartley, J. Chem. Soc. (London), **27**, 501 (1874) *48*

$CoBr_2 \cdot 6H_2O$

NaOH **III-23**

When hexa-hydrated cobaltous bromide is boiled with sodium hydroxide, cobaltous hydroxide and sodium bromide are formed.

$$CoBr_2 \cdot 6H_2O + 2NaOH \rightarrow Co(OH)_2 + 2NaBr + 6H_2O$$

Walter N. Hartley, J. Chem. Soc. (London), **27**, 501 (1874) *48*

$CoBr_2 \cdot 6H_2O$

Δ **III-24**

When the hexa-hydrated cobaltous bromide is heated to 100°, four molecules of water are given off and the di-hydrated cobaltous bromide is formed.

(a) $CoBr_2 \cdot 6H_2O \rightarrow CoBr_2 \cdot 2H_2O + 4H_2O$

(b) $CoBr_2 \cdot 2H_2O + \Delta \rightarrow CoBr_2 + 2H_2O$

Walter N. Hartley, J. Chem. Soc. (London), **27**, 502 (1874) *48*
Ref., R. Gerstl, Ber., **7**, 739 (1874) *11*

$CoBr_2 \cdot 6H_2O$

Decomp. **III-25**

P

If hexa-hydrated cobaltous bromide is kept in vacuo over sulfuric acid, it will be slowly dehydrated to the anhydrous, green cobaltous bromide. The dehydration requires several days.

$$CoBr_2 \cdot 6H_2O \rightarrow CoBr_2 + 6H_2O$$

Walter N. Hartley, J. Chem. Soc. (London), **27**, 502 (1874) *48*

$CoBrCO_3 \cdot en_2$

HNO_3 **III-26**

NaBr

To 30 g. diethylenediamine cobalt (III) bromide carbonate in 120 cc. cold water is added 15 cc. concentrated nitric acid. After adding slightly more potassium hydroxide solution than is required for neutralization and some solid sodium bromide, a red precipitate of diethylenediamine cobalt dibromide monohydroxo hydrate is formed.

(a) $CoBrCO_3 \cdot 2C_2H_4(NH_2)_2 + 2HNO_3$
$\rightarrow (H_2O + CO_2 + CoBr(NO_3)_2 \cdot 2C_2H_4(NH_2)_2)$

(b) $CoBr(NO_3)_2 \cdot 2C_2H_4(NH_2)_2 + KOH$
$\rightarrow (KNO_3 + CoBrNO_3OH \cdot 2C_2H_4(NH_2)_2)$

(c) $CoBrNO_3OH \cdot 2C_2H_4(NH_2)_2 + H_2O + NaBr$
$\rightarrow \underline{(NaNO_3) + CoBr_2OH \cdot 2C_2H_4(NH_2)_2 \cdot H_2O}$

A. Werner and S. Matissen, Helv. Chim., **1**, 79 (1918) *78*

$CoBr_2C_5H_7O_2 \cdot en_2 \cdot H_2O$

AgCl **III-27**

An aqueous solution of diethylenediamine cobalt dibromide monoacetylacetonate hydrate is shaken with freshly precipitated silver chloride. The solution is then filtered, concentrated to a sirupy consistency, and allowed to stand, whereupon crystals of diethylenediamine cobalt dichloride monoacetylacetonate dihydrate precipitate.

$CoBr_2C_5H_7O_2 \cdot 2C_2H_4(NH_2)_2 \cdot H_2O + 2AgCl + H_2O$
$\rightarrow \underline{CoCl_2C_5H_7O_2 \cdot 2C_2H_4(NH_2)_2 \cdot 2H_2O} + \underline{2AgBr}$

A. Werner and S. Matissen, Helv. Chim., **1**, 80 (1918) *78*

$CoBr_2C_5H_7O_2 \cdot en_2 \cdot H_2O$

$AgNO_3$ **III-28**

To a concentrated aqueous solution of diethylenediamine cobalt dibromide monoacetylacetonate hydrate is added a solution of silver nitrate. The solution is filtered, the excess silver in the filtrate precipitated with sodium chloride, the solution again filtered, concentrated, and allowed to stand, whereupon crystals of diethylenediamine cobalt dinitrate monoacetylacetonate precipitate.

$CoBr_2C_5H_7O_2 \cdot 2C_2H_4(NH_2)_2 \cdot H_2O + 2AgNO_3$
$\rightarrow \underline{Co(NO_3)_2 \cdot C_5H_7O_2 \cdot 2C_2H_4(NH_2)_2} + \underline{2AgBr} + (H_2O)$

A. Werner and S. Matissen, Helv. Chim., **1**, 80, (1918) *78*

$CoBr_2C_5H_7O_2 \cdot en_2 \cdot H_2O$

Ag_2SO_4 **III-29**

An aqueous solution of diethylenediamine cobalt dibromide monoacetylacetonate hydrate is shaken with freshly precipitated silver sulfate and filtered. The excess silver is precipitated with sodium chloride, the solution again filtered, concentrated, and allowed to stand, whereupon crystals of diethylenediamine cobalt sulfate acetylacetonate dihydrate precipitate.

$$CoBr_2C_5H_7O_2 \cdot 2C_2H_4(NH_2)_2 \cdot H_2O + Ag_2SO_4 + H_2O \rightarrow \underline{CoSO_4C_5H_7O_2 \cdot 2C_2H_4(NH_2)_2 \cdot 2H_2O} + \underline{2AgBr}$$

A. Werner and S. Matissen, Helv. Chim., **1**, 81 (1918) *78*

$CoBr_2C_5H_7O_2 \cdot en_2 \cdot H_2O$

$HClO_4$ **III-30**

A concentrated aqueous solution of diethylenediamine cobalt dibromide monoacetylacetonate hydrate is treated with perchloric acid or solid sodium perchlorate to give a precipitate of diethylenediamine cobalt diperchlorate monoacetylacetonate.

$$CoBr_2C_5H_7O_2 \cdot 2C_2H_4(NH_2)_2 \cdot H_2O + 2HClO_4 \rightarrow \underline{Co(ClO_4)_2C_5H_7O_2 \cdot 2C_2H_4(NH_2)_2} + (2HBr + H_2O)$$

A. Werner and S. Matissen, Helv. Chim., **1**, 82 (1918) *78*

$CoBr_2C_5H_7O_2 \cdot en_2 \cdot H_2O$

KI **III-31**

A solution of diethylenediamine cobalt dibromide monoacetylacetonate hydrate is triturated with solid potassium iodide to produce a bright red precipitate of diethylenediamine cobalt di-iododimonoacetylacetonate hydrate.

$$CoBr_2C_5H_7O_2 \cdot 2C_2H_4(NH_2)_2 \cdot H_2O + 2KI \rightarrow \underline{CoI_2C_5H_7O_2 \cdot 2C_2H_4(NH_2)_2 \cdot H_2O} + (2KBr)$$

A. Werner and S. Matissen, Helv. Chim., **1**, 80 (1918) *78*

$CoBr_2C_7H_6NO_2 \cdot en_2$

$HClO_4$ **III-32**

Diethylenediamine cobalt dibromide monobenzohydroxamate reacts with perchloric acid to give diethylenediamine cobalt diperchlorate monobenzohydroxamate.

$$CoBr_2C_7H_6NO_2 \cdot 2C_2H_4(NH_2)_2 + 2HClO_4 \rightarrow Co(ClO_4)_2 \cdot C_7H_6NO_2 \cdot 2C_2H_4(NH_2)_2 + (2HBr)$$

A. Werner and S. Matissen, Helv. Chim., **1**, 83 (1918) *78*

$CoBr_2C_7H_6NO_2 \cdot en_2$

NH_4SCN **III-33**

To a concentrated aqueous solution of diethylenediamine cobalt dibromide monobenzohydroxamate is added ammonium thiocyanate to obtain an amorphous precipitate of diethylenediamine cobalt dithiocyanate monobenzohydroxamate which crystallizes on standing.

$$CoBr_2C_7H_6NO_2 \cdot 2C_2H_4(NH_2)_2 + 2NH_4SCN \rightarrow \underline{Co(SCN)_2C_7H_6NO_2 \cdot 2C_2H_4(NH_2)_2} + (2NH_4Br)$$

A. Werner and S. Matissen, Helv. Chim., **1**, 83 (1918) *78*

$CoBr_2C_7H_6NO_2 \cdot en_2$

$(NH_4)_2SO_4$ **III-34**

A concentrated aqueous solution of diethylenediamine cobalt dibromide monobenzohydroxamate is triturated with solid ammonium sulfate to obtain a precipitate of diethylenediamine cobalt monosulfate monobenzohydroxamate trihydrate.

$$CoBr_2C_7H_6NO_2 \cdot 2C_2H_4(NH_2)_2 + (NH_4)_2SO_4 + 3H_2O \rightarrow \underline{CoSO_4C_7H_6NO_2 \cdot 2C_2H_4(NH_2)_2 \cdot 3H_2O} + (2NH_4Br)$$

A. Werner and S. Matissen, Helv. Chim., **1**, 84 (1918) *78*

$CoBr_2C_7H_6NO_2 \cdot en_2$

$Na_2S_2O_6$ **III-35**

An aqueous solution of diethylenediamine cobalt dibromide monobenzohydroxamate is triturated with solid sodium dithionate to obtain a precipitate of diethylenediamine cobalt dithionate monobenzohydroxamate dihydrate.

$$CoBr_2C_7H_6NO_2 \cdot 2C_2H_4(NH_2)_2 + Na_2S_2O_6 + 2H_2O \rightarrow \underline{CoS_2O_6C_7H_6NO_2 \cdot 2C_2H_4(NH_2)_2 \cdot 2H_2O} + (2NaBr)$$

A. Werner and S. Matissen, Helv. Chim., **1**, 83 (1918) *78*

$CoBr_2OH \cdot en_2.H_2O$

Acetyl Acetone **III-36**

One g. diethylenediamine cobalt dibromide monohydroxo hydrate dissolved in 10 cc. water is refluxed for half an hour with 0.6 g. acetyl acetone. The solution is then concentrated to half its volume and sodium or potassium bromide added, whereupon red crystals of diethylenediamine cobalt dibromide monoacetylacetonate hydrate precipitate.

$$CoBr_2OH \cdot 2C_2H_4(NH_2)_2 \cdot H_2O + CH_2(COCH_3)_2 \rightarrow \underline{CoBr_2C_5H_7O_2 \cdot 2C_2H_4(NH_2)_2 \cdot H_2O} + (H_2O)$$

A. Werner and S. Matissen, Helv. Chim., **1**, 79 (1918) *78*

$CoBr_2OH \cdot en_2 \cdot H_2O$

Benzohydroxamic acid **III-37**

An aqueous solution of diethylenediamine cobalt dibromide monohydroxo hydrate is refluxed with benzohydroxamic acid for half an hour. Addition of solid potassium or sodium bromide precipitates diethylenediamine cobalt dibromide monobenzohydroxamate.

$$CoBr_2OH \cdot 2C_2H_4(NH_2)_2 \cdot H_2O + C_6H_5COHNOH \rightarrow \underline{CoBr_2C_7H_6NO_2 \cdot 2C_2H_4(NH_2)_2} + (2H_2O)$$

A. Werner and S. Matissen, Helv. Chim., **1**, 82 (1918) *78*

$Co(CH_3COO)_2$

Na_2Te **III-38**

$HC_2H_3O_2$

A heavy black precipitate forms when a solution of cobalt acetate, acidified with acetic acid is treated with sodium telluride.

$$2Co(C_2H_3O_2)_2 + 3Na_2Te + 4H_2O + 2HC_2H_3O_2 \rightarrow \underline{Co_2Te_3 \cdot 4H_2O} + (6NaC_2H_3O_2 + H_2)$$

C. A. Tibbals, Jr., J. Am. Chem. Soc., **31**, 908 (1909) *1*

$Co(CH_3COO)_2$

ε **III-39**

A solution of cobaltous acetate if subjected to electrolysis is oxidized to the trivalent cobalt salt. This decomposes with water again to the divalent state and liberates oxygen.

(a) $Co(CH_3COO)_2 + CH_3COO^- \rightarrow Co(CH_3COO)_3 + (^-)$

(b) $4Co(CH_3COO)_3 + 2H_2O \rightarrow 4Co(CH_3COO)_2 + 4CH_3COOH + O_2$

K. Elbs, Z. Elektrochem., **3**, 70 (1896) *86*

$Co(CH_3COO)_2 \cdot 4H_2O$

CH_3COOH **III-40**

$NaNO_2$

Sodium cobaltinitrite solution can be prepared by dissolving cobalt acetate in water and acetic acid, then dissolving sodium nitrite in water, filtering and mixing the two solutions.

$$(Co(C_2H_3O_2)_2 \cdot 4H_2O + CH_3COOH + 6NaNO_2 \rightarrow Na_3Co(NO_2)_6 + 3NaC_2H_3O_2 + 4H_2O + (H))$$

R. H. Adie and T. B. Wood, J. Chem. Soc. (London), **77**, 1076 (1900) *120*

$Co(CH_3COO)_2 \cdot 4H_2O$

$NaNO_2$ **III-41**

CH_3COOH

The solutions of cobalt acetate, glacial acetic acid and sodium nitrite are added together and gently warmed. NO is evolved and evacuated, the following reaction takes place.

$$14NaNO_2 + 2Co(C_2H_3O_2)_2 \cdot 4H_2O + 4HC_2H_3O_2 \rightarrow 8NaC_2H_3O_2 + Na_6Co_2(NO_2)_{12} \cdot 2H_2O + 2NO + 6H_2O + 2H_2$$

Bowser, J. Ind. Eng. Chem., **1**, 791 (1909) *22*

$Co(CH_3COO)_3$

$C_{10}H_6ONO$ **III-42**

Cobaltic acetate will react with a 2% solution of α-nitroso β-naphthol in acetic acid yielding a precipitate of an inner complex cobalt salt.

$$Co(CH_3COO)_3 + 3C_{10}H_7ONO + 2H_2O \rightarrow \underline{Co(C_{10}H_6ONO)_3 \cdot 2H_2O} + 3HC_2H_3O_2$$

C. Mayr and F. Feigl., Z. anal. Chem., **90**, 17 (1932) *28*

$Co(CH_3COO)_3$

$C_{10}H_7ONO_2$ **III-43**

Cobaltic acetate will react with α-nitro β-naphthol yielding an insoluble cobalt salt.

$$Co(CH_3COO)_3 + 3C_{10}H_7ONO_2 \rightarrow \underline{Co(C_{10}H_6ONO_2)_3} + 3HC_2H_3O_2$$

C. Mayr, Z. anal. Chem., **98**, 402 (1934) *28*

$Co(CH_3COO)_3$

Δ **III-44**

An aqueous solution of cobaltic acetate changes to the trioxide when heated to 170°.

$$2Co(C_2H_3O_2)_3 + 15H_2O \rightarrow (12CO_2 + 24H_2) + Co_2O_3$$

Riban,
Ref., A. Henninger, Ber., **9:2**, 1133 (1876) *26*

$Co(CN)_2$

KCN **III-45**

Warm solutions of cobaltous cyanide and potassium cyanide are added together while air is excluded.

$$2Co(CN)_2 + 8KCN + 2H_2O \rightarrow \underline{2K_2Co(CN)_6} + 2KOH + H_2$$

Zwenger, Ann., **62**, 162 (1847) *25*

$Co(CNS)_2$

AgCNS **III-46**

A very conc. solution of cobalt thiocyanate was saturated with solid silver thiocyanate and cooled. The salt formed, has a brilliant blue color; it loses water below 100° and its color changes to dark green.

$$Co(CNS)_2 + AgCNS + 2H_2O \rightarrow CoAg(CNS)_3 \cdot 2H_2O$$

Shinn and Wells, Am. Chem. J., **29**, 477 (1903) *21*

$Co(CNS)_2$

CSN_2H_4 **III-47**

Thiourea is added to a diluted solution of cobalt thiocyanate. The liquid is kept boiling for a short time. On cooling, brown colored needles are formed.

$$Co(CNS)_2 + 2CSN_2H_4 \rightarrow \underline{Co(CSN_2H_4)_2(CNS)_2}$$

A. Rosenheim and V. J. Meyer, Z. anorg. Chem., **49**, 13 (1906) *28*

$CoCO_3$

Br_2 **III-48**

Na_2CO_3

Solutions of cobalt carbonate, bromine, and sodium carbonate probably react according to the following equations.

(a) $Br_2 + 2Na_2CO_3 + H_2O \rightarrow 2NaBr + 2NaHCO_3 + O$

(b) $4NaHCO_3 + 2CoCO_3 + O \rightarrow [Co(NaCO_3)_2]_2O + 2CO_2 + 2H_2O$

R. B. Durrant, J. Chem. Soc. (London), **87**, 1790 (1905) *13*
Ref., Green, Proc. Roy. Soc. Edin., **7**, 56 (1890)

$CoCO_3$

$CH_2ClCOOH$ **III-49**

CCl_3COOH

Cobalt carbonate will react with monochloroacetic acid yielding cobaltous monochloroacetate of syrup-like consistency. The same type reaction occurs with trichloroacetic acid.

(a) $CoCO_3 + 2CH_2ClCOOH \rightarrow \underline{Co(CH_2ClCOO)_2} + H_2O + CO_2$

(b) $CoCO_3 + 2CCl_3COOH + 3H_2O$
$\rightarrow \underline{Co(CCl_3COO)_2 \cdot 4H_2O} + CO_2$

F. Reitzenstein, Z. anorg. Chem., **32**, 298 (1902) *28*

$CoCO_3$

$CH_2SHCOOH$ **III-50**

$CH_2SHCOONa$

Thioglycolic acid is saturated at 100° with cobalt carbonate. A precipitate of brownish red color, the acid cobaltous salt of the complex cobaltous-thioglycolic acid is formed. A similar type reaction occurs with sodium thioglycolate.

(a) $3CoCO_3 + 2CH_2SHCOOH + 12H_2O$
$\rightarrow (Co(SCH_2COO)_2)_2CoH_2 \cdot 11H_2O + 3CO_2$

(b) $CoCO_3 + 2CH_2SHCOONa + 5H_2O$
$\rightarrow Co(SCH_2COONa)_2 \cdot 6H_2O + CO_2$

A. Rosenheim and I. Davidsohn, Z. anorg. Chem., **41**, 231 (1904) *28*

$CoCO_3$

$HClO_4$ **III-51**

Cobaltous carbonate is treated with perchloric acid and evaporated in a vacuum over sulfuric acid.

$$CoCO_3 + 2HClO_4 \rightarrow Co(ClO_4)_2 + (H_2O + CO_2)$$

Goldblum and Terlikowski, Bull. soc. chim. [4] (1912) (France) **11**, 103 *25*

$CoCO_3$

HIO_3 **III-52**

Cobalt (II) carbonate, when dissolved in warm iodic acid, is transformed into cobalt (II) iodate.

$$CoCO_3 + 2HIO_3 \rightarrow Co(IO_3)_2 + (H_2O + CO_2)$$

A. Werner and P. Karrer, Helv. Chim., **1**, 62 (1918) *78*

$CoCO_3$

HNO_3 **III-53**

If cobalt carbonate be treated with dilute nitric acid, cobalt nitrate will be formed; carbon dioxide and water will be formed at the same time.

$$CoCO_3 + 2HNO_3 \rightarrow Co(NO_3)_2 + H_2O + CO_2$$

Samuel H. C. Briggs, J. Chem. Soc. (London), **115:1**, 75 (1919) *48*

$CoCO_3$

$H_2C_4H_4O_5$ **III-54**

Cobalt carbonate dissolves when digested with malic acid, and on treatment with alcohol, a basic cobaltomalate (amethyst colored) is precipitated. The pentahydrate forms at 100°C and the tetrahydrate at 140°C.

$$2CoCO_3 + H_2C_4H_4O_5 + 4H_2O \rightarrow CoC_4H_4O_5 \cdot CoO \cdot 5H_2O + (2CO_2)$$

S. U. Pickering, J. Chem. Soc. (London), **107**, 946 (1915) *57*

$CoCO_3$

H_2CrO_4 **III-55**

Cobalt chromate is formed when cobalt carbonate is heated with chromic acid.

$$CoCO_3 + H_2CrO_4 \rightarrow CoCrO_4 + (H_2O + CO_2)$$

S. H. C. Briggs, Z. anorg. Chem., **63**, 325 (1909)
Ref., J. L. Howe, J. Am. Chem. Soc., **33**, 183 (1911) *1*

$CoCO_3$

H_2F_2 **III-56**

Cobalt fluoride is formed when cobalt carbonate is treated with hydrofluoric acid.

$$CoCO_3 + H_2F_2 \rightarrow CoF_2 + H_2O + CO_2$$

Berzelius, Ann. chim. phys. [2], **24**, 61 (1823)
Ref., ibid., Pogg. Ann., **1**, 28 (1824)
Ref., Edmister and Cooper, J. Am. Chem. Soc., **42**, 2419 (1920) *1*

$CoCO_3$

H_2F_2 **III-57**

Cobaltous carbonate is dissolved in hydrofluoric acid.

$$2CoCO_3 + 3H_2F_2 + 12H_2O \rightarrow 2CoF_2 \cdot HF \cdot 6H_2O + (2H_2CO_3)$$

F. H. Edmister and H. C. Cooper, Science, **50**, 444 (1919) *25*

$CoCO_3$

H_2O_2 **III-58**

Cobaltous carbonate is treated with hydrogen peroxide in the presence of potassium acid carbonate.

$$2CoCO_3 + H_2O_2 \rightarrow Co_2O(CO_3)_2 + H_2O$$

R. G. Durrant, J. Chem. Soc. (London), **87**, 1782 (1905) *25*

$CoCO_3$

H_2O_2 **III-59**

$KHCO_3$

If cobalt carbonate is reacted with hydrogen peroxide and potassium bicarbonate, the products are carbon dioxide, water and cobalt potassium carbonate.

$$4KHCO_3 + 2CoCO_3 + H_2O_2 \rightarrow [Co(KCO_3)_2]_2O + 2CO_2 + 3H_2O$$

R. G. Durrant, J. Chem. Soc. (London), **87**:2, 1783 (1905) *103*

$CoCO_3$

H_2SO_3 **III-60**

Cobaltous carbonate is dissolved in a solution of sulfurous acid and the solution treated with alcohol; the product crystallizes with water.

$$CoCO_3 + H_2SO_3 + 4H_2O \rightarrow CoSO_3 \cdot 5H_2O + (CO_2)$$

Rohrig, J. f. prakt. Chem. [2], **37**, 217 (1888) *25*

$CoCO_3$

H_2SO_4 **III-61**

Sulfuric acid reacts with cobalt carbonate to form cobalt sulfate.

$$CoCO_3 + H_2SO_4 \rightarrow CoSO_4(+H_2O + CO_2)$$

H. H. Denham, J. Chem. Soc. (London), **93**, 61 (1908) *57*

$CoCO_3$

H_2SO_4 **III-62**

Cobaltous carbonate is dissolved in dilute sulfuric acid and the filtrate concentrated; the product crystallizes with $7H_2O$.

$$CoCO_3 + H_2SO_4 + 6H_2O \rightarrow CoSO_4 \cdot 7H_2O + CO_2$$

Oechsner de Coninck, Bull. Acad. (Belg.), 633 (1904) *25*

$CoCO_3$

H_2SeO_3 **III-63**

A cold solution of cobaltous carbonate is treated with an equal volume of selenous acid in water and heated.

$$3CoCO_3 + 3H_2SeO_3 \rightarrow (CoSeO_3)_3 \cdot H_2O + 2H_2O + 3CO_2$$

Bourtzoureano, Ann. chim. phys. [6], **18**, 318 (1889) *25*

$CoCO_3$

H_3PO_4 **III-64**

Cobaltous carbonate is dissolved in phosphoric acid and the solution boiled.

$$2CoCO_3 + 2H_3PO_4 + H_2O \rightarrow 2CoHPO_4 \cdot 3H_2O + 2CO_2$$

Debray, Ann. chim, phys. [3], **61**, 438 (1860) *25*

$CoCO_3$

KCN **III-65**

O_2

$C_2O_2H_4$

Potassium cobalti-cyanide is prepared by dissolving cobaltous carbonate in acetic acid, then adding a solution of potassium cyanide and oxidizing with a current of air:

(a) $(CoCO_3 + 2C_2O_2H_4 \rightarrow Co(C_2O_2H_3)_2 + CO_2 + H_2O)$

(b) $(4Co(C_2O_2H_3)_2 + 24KCN + 2H_2O + O_2 \rightarrow 4K_3[Co(CN)_6] + 8KC_2H_3O_2 + 4KOH)$

Christensen, J. f. prakt. Chem., **31**, 163 (1885)
Ref., Mathieu and Cornevin, J. Chim. phys., **36**, 271 (1939) *69*

$CoCO_3$

$KHCO_3$ **III-66**

H_2O_2

Cobalt carbonate, potassium bicarbonate, and hydrogen peroxide react in solution to form $[Co(KCO_3)_2]_2O$, carbon dioxide and water.

$$4KHCO_3 + 2CoCO_3 + H_2O_2 \rightarrow (Co(KCO_3)_2)_2O + 2CO_2 + 3H_2O$$

R. G. Durrant, J. Chem. Soc. (London), **87**, 1781 (1905) *13*

$CoCO_3$

HNO_2

KNO_2 **III-67**

To a suspension of cobaltous carbonate in a small amount of water is added potassium nitrite and nitrous acid yielding potassium cobaltinitrite and potassium carbonate.

$$2CoCO_3 + 10KNO_2 + 2HNO_2 \rightarrow 2K_3Co(NO_2)_6 + 2K_2CO_3 + H_2$$

Rosenheim and Koppel, Z. anorg. Chem., **17**, 42 (1898) *13*

$CoCO_3$

N_3H **III-68**

Cobalt carbonate will react with triazoic acid yielding cobalt triazide, a powder of reddish-brown color.

$$CoCO_3 + 2N_3H \rightarrow CoN_6 + H_2O + CO_2$$

Lothar Wöhler and F. Martin, Ber., **50**, 586 (1917) *28*

$CoCO_3$

$S(CH_2COOH)_2$ **III-69**

$C_2H_4(NH_2)_2$

Mixture of cobalt carbonate solution with thiodiacetic acid and ethylenediamine results in the formation of a clear solution. After evaporating and cooling, a double salt precipitates.

$$CoCO_3 + 2S(CH_2COOH)_2 + C_2H_4(NH_2)_2 + H_2O$$
$$\rightarrow \underline{S(CH_2COO)_2Co \cdot S(CH_2COOH)_2 \cdot C_2H_4(NH_2)_2 \cdot 2H_2O} + (CO_2)$$

T. S. Price and S. A. Brazier, J. Chem. Soc. (London), **107**, 1373 (1915) *57*

$CoCO_3$

SO_2 **III-70**

Cobaltous carbonate solution is saturated with sulfur dioxide. The product crystallizes with $3H_2O$.

$$CoCO_3 + SO_2 + 4H_2O \rightarrow CoSO_3 \cdot 3H_2O + (H_2CO_3)$$

Schulz, Tids. Kemi. Bergvaesen, **4**, 195 (1924) *25*

$CoCO_3NO_3 \cdot 4NH_3$

HNO_3 **III-71**

If a solution of carbonato cobaltic nitrate tetrammoniate is treated with dilute nitric acid, warmed gently and just neutralized with potassium hydroxide, dihydrated cobaltic nitrate tetrammoniate will be obtained in the solution.

$$CoCO_3NO_3 \cdot 4NH_3 + 2HNO_3 + H_2O$$
$$\rightarrow Co(NO_3)_3 \cdot 2H_2O \cdot 4NH_3 + CO_2$$

Samuel H. C. Briggs, J. Chem. Soc. (London), **115**:1, 72 (1919) *48*

$Co(CO)_4$

Br_2 **III-72**

Cobalt tetracarbonyl reacts with bromine to yield cobaltous bromide and carbon monoxide.

$$Co(CO)_4 + Br_2 \rightarrow CoBr_2 + 4CO$$

L. Mond, H. Hirtz, and M. D. Cowap, J. Chem. Soc. (London), **97**:1, 804 (1910) *108* *13*

$Co(CO)_4$

Br_2 **III-73**

HNO_3

Cobalt carbonyl is decomposed in presence of nitric acid or bromine.

(a) $$Co(CO)_4 + Br_2 \rightarrow CoBr_2 + 4CO$$

(b) $$2Co(CO)_4 + 6HNO_3 \rightarrow 2Co(NO_3)_2 + 8CO + (3H_2O + NO + NO_2)$$

L. Mond, H. Hirtz and M. D. Cowap, J. Chem. Soc. (London), **97**, 804 (1910) *57*

$Co(CO)_4$

NO **III-74**

Nitric oxide reacts with cobalt tetracarbonyl at 40°.

$$Co(CO)_4 + NO \rightarrow Co(CO)_3 \cdot NO + CO$$

Mond and Wallis, J. Chem. Soc. (London), **121**, 29 (1921) *25*

$Co(CO)_4$

Δ **III-75**

Cobalt tetracarbonyl is decomposed to tricarbonyl at 60° in about two days.

$$Co(CO)_4 \rightarrow Co(CO)_3 + CO$$

L. Mond, H. Hirtz and M. D. Cowap, J. Chem. Soc. (London), **97**, 804 (1910) *57* *13*

Acc. no:- 21099

CoC_2O_4

KOH **III-76**

Normal cobalt oxalate is suspended in water, the air excluded and then treated with potassium hydroxide.

$$3CoC_2O_4 + 4KOH \rightarrow \underline{2Co(OH)_2 \cdot CoC_2O_4} + 2K_2C_2O_4$$

C. Winkelblech, Ann., **13**, 158 (1835) *25*

CoC_2O_4

$K_2C_2O_4$ **III-77**

Cobalt oxalate is dissolved by a solution of potassium oxalate and the solution evaporated.

$$3CoC_2O_4 + 2K_2C_2O_4 \rightarrow (K_2C_2O_4)_2 \cdot (CoC_2O_4)_3$$

Deakin, Scott and Steele, Z. physik. Chem., **69**, 123 (1909) *25*

CoC_2O_4

Δ **III-78**

CO

H_2

Cobalt oxalate is converted to oxide by heating. Cobalt oxide is reduced to cobalt by hydrogen at 300° and 5 atmospheres pressure. Cobalt thus prepared, heated with carbon monoxide at 150° and 30–40 atmospheres, forms cobalt carbonyl.

(a) $$CoC_2O_4 \rightarrow CoO + CO_2 + CO$$

(b) $$CoO + H_2 \rightarrow Co + H_2O$$

(c) $$Co + 4CO \rightarrow Co(CO)_4$$

L. Mond, H. Hirtz and M. D. Cowap, J. Chem. Soc. (London), **97**, 803 (1910) *57*

$CoC_3H_2O_4$

$K_2C_3H_2O_4$ **III-79**

A double malonate of cobalt and potassium can be prepared by dividing a sample of malonic acid into two equal fractions, and neutralizing the first half with potassium hydroxide and the other with a slurry of cobalt hydroxide. Mix the two solutions, add a little malonic acid and evaporate on a water bath. The hydrated double salt of a pinkish tinge will deposit. Anhydrous potassium cobaltomalonate is formed by complete evaporation of the solution. The same type reactions occur when the malonates of ammonium rubidium and cesium are mixed with cobalt malonate.

$$CoC_3H_2O_4 + K_2C_3H_2O_4 + 4H_2O \rightarrow K_2Co(C_3H_2O_4)_2 \cdot 4H_2O$$

R. C. Lord, J. Phys. Chem., **11**, 173 (1907) *7*

$Co(C_5H_5N)_4SiF_6$

Δ **III-80**

Cobalt tetrapyridine fluosilicate (from the reaction of a water solution of the fluosilicate and pyridine) decomposes at temperatures up to 83° in the following manner. (At higher temperatures silicon tetrafluoride is given off.)

(a) $Co(C_5H_5N)_4SiF_6 \rightarrow Co(C_5H_5N)_3SiF_6 + C_5H_5N$

(b) $Co(C_5H_5N)_3SiF_6 \rightarrow Co(C_5H_5N)_2SiF_6 + C_5H_5N$

W. T. L. Broeck and P. A. Vander Meulen, J. Am. Chem. Soc., **53**, 3599 (1931) *16*

$Co(C_{18}H_{33}O_2)_2$

Cu **III-81**

Pb

Cobalt oleate in toluene reacts at the boiling point with metallic copper and with lead which become coated with a white lustrous and a black deposit respectively. In the same way lead reacts with solutions of anhydrous cadmium oleate, chromic oleate, manganous oleate and nickel oleate in toluene at the boiling point. There is no reaction with magnesium, cadmium, zinc, bismuth, antimony, cobalt, aluminum, silver, platinum, iron or copper. Ferrous oleate under the same conditions reacts with none of the metals.

(a) $Co(C_{18}H_{33}O_2)_2 + Cu \rightarrow Co + Cu(C_{18}H_{33}O_2)_2$

(b) $Co(C_{18}H_{33}O_2)_2 + Pb \rightarrow Co + Pb(C_{18}H_{33}O_2)_2$

J. L. Sammis, J. Phys. Chem., **10**, 593 (1906) *7*

$Co(C_{18}H_{33}O_2)_2$

HCl **III-82**

Dry hydrogen chloride, when added to a solution of cobalt oleate in benzene, causes an immediate precipitation of blue cobalt chloride. The reaction proceeds quantitatively but the precipitate is contaminated by some oleic acid. When nickel oleate is substituted for the cobalt salt, a similar reaction occurs, brownish yellow nickel chloride being formed. With copper oleate solutions similar behavior is observed, the precipitate consisting of brown copper chloride. The reactions are not ionic.

(a) $Co(C_{18}H_{33}O_2)_2 + 2HCl \rightarrow 2C_{18}H_{33}O_2H + \underline{CoCl_2}$

(b) $Ni(C_{18}H_{33}O_2)_2 + 2HCl \rightarrow 2C_{18}H_{33}O_2H + \underline{NiCl_2}$

(c) $Cu(C_{18}H_{33}O_2)_2 + 2HCl \rightarrow 2C_{18}H_{33}O_2H + \underline{CuCl_2}$

L. Kahlenberg, J. Phys. Chem., **6**, 1 (1902) 7

$Co(C_{18}H_{33}O_2)_2$

H_2S **III-83**

Dry hydrogen sulfide gas passed into a solution of cobalt oleate in dry benzene will precipitate the metal sulfide at once. Similar reactions are observed when hydrogen sulfide is passed into solutions of nickle or copper oleate in benzene.

(a) $Co(C_{18}H_{33}O_2)_2 + H_2S \rightarrow 2C_{18}H_{33}O_2H + \underline{CoS}$

(b) $Ni(C_{18}H_{33}O_2)_2 + H_2S \rightarrow 2C_{18}H_{33}O_2H + \underline{NiS}$

(c) $Cu(C_{18}H_{33}O_2)_2 + H_2S \rightarrow 2C_{18}H_{33}O_2H + \underline{CuS}$

L. Kahlenberg, J. Phys. Chem., **6**, 1 (1902) 7

$CoClBr_2(H_2O)\cdot 3NH_3$

H_2O **III-84**

Trihydroxo dicobalt tribromide hexammine is prepared by warming aquocobalt chlorodibromide triammine to 60°.

$$2[CoClBr_2(H_2O)\cdot 3NH_3] + H_2O \rightarrow Co_2(OH)_3Br_3\cdot 6NH_3 + (2HCl] + [HBr)$$

A. Werner, Ber., **40**, 4834 (1907)
Ref., J. J. Sudborough, J. Chem. Soc. (London), **94:2**, 43 (1908) 57

$CoClCrO_4(H_2O)_5 \cdot 5NH_3$

$AgNO_3$ **III-85**

A solution of hydrated chromato cobaltic chloride pentammoniate, when treated with silver nitrate, yields a red precipitate of silver chromate containing traces of silver chloride. Faintly colored hydrated cobaltic chloro nitrate pentammoniate remains in solution.

$$CoClCrO_4(H_2O)_5 \cdot 5NH_3 + 2AgNO_3 \rightarrow \underline{Ag_2CrO_4} + CoCl(NO_3)_2 \cdot (H_2O)_5 \cdot 5NH_3$$

Samuel H. C. Briggs, J. Chem. Soc. (London), **115**:I, 71 (1919) *48*

$Co(ClO_3)_2$

NH_3 **III-86**

A concentrated solution of cobalt chlorate, which is kept in a hydrogen atmosphere, is treated with a solution of concentrated ammonia. Unstable crystals of pale red color are formed.

$$Co(ClO_3)_2 + 6NH_3 \rightarrow \underline{Co(ClO_3)_2 \cdot 6NH_3}$$

F. Ephraim and A. Jansen, Ber., **48**, 46 (1915) *28*

$Co(ClO_4)_2 \cdot 6NH_3$

H_2O **III-87**

By heating the salt cobalt perchlorate hexammoniate with water the result will be cobalt hydroxide, cobalt perchlorate, ammonium perchlorate and ammonia.

$$2Co(ClO_4)_2 \cdot 6NH_3 + 2H_2O \rightarrow Co(OH)_2 + Co(ClO_4)_2 + 2NH_4ClO_4 + 4NH_3$$

Roberto Salvadori, Gazz. chim. ital., **40**, 17 (1910) *21*

$CoCl_2$

$Ba(CN)_2$ **III-88**

Cobaltous chloride in solution reacts with barium cyanide.

$$CoCl_2 + 3Ba(CN)_2 \rightarrow Ba_2Co(CN)_6 + BaCl_2$$

Descamps, Ann. chim. phys. [5], **24**, 178, (1881) *25*

$CoCl_2$

CO **III-89**

KOH

Carbon monoxide is absorbed by an alkaline suspension of a cobalt salt to form $KCo(CO)_4$ when a carrier such as a small amount of a cyanide or cysteine is present.

$$2CoCl_2 + 11CO + 12KOH \rightarrow 3K_2CO_3 + 2KCo(CO)_4 + 4KCl$$

A. A. Blanchard and P. Gilmont, J. Am. Chem. Soc., **62**, 1192 (1940) *16*

$CoCl_2$

CSN_2H_4 **III-90**

Blue-black colored crystals are obtained from the slow crystallizing of a solution containing 1 mole of cobaltous chloride and 2 moles of thiourea.

$$2CoCl_2 + 7CSN_2H_4 \rightarrow \underline{Co_2(CSN_2H_4)_7Cl_4}$$

A. Rosenheim and V. J. Meyer, Z. anorg. Chem., **49**, 13 (1906) *28*

$CoCl_2$

$C_3H_{10}ON_2$ **III-91**

With cobaltous salts 1:3 diamino isopropanol forms cobaltic coordination compounds which can also be obtained with roseo-cobaltic salts.

$$(2CoCl_2 + 2C_3H_{10}ON_2 \rightarrow 2Co(C_3H_9ON_2)Cl + 2HCl)$$

F. G. Mann, J. Chem. Soc. (London), 2904 (1927).
Ref., J. G. Breckenridge and J. W. R. Hodgins, Can. J. Res., **B17**, 331 (1939) *69*

$CoCl_2$

C_5H_5N **III-92**

Crystals of deep-red color are obtained when a solution of cobalt chloride reacts with pyridine at elevated temperature. By heating the red crystals for one hour at a temperature of 115°–120° partial decomposition takes place.

(a) $$CoCl_2 + 4C_5H_5N \rightarrow \underline{CoCl_2 \cdot 4C_5H_5N}$$

(b) $$CoCl_2 \cdot 4C_5H_5N \rightarrow \underline{CoCl_2 \cdot C_5H_5N} + 3C_5H_5N$$

F. Reizenstein, Z. anorg. Chem., **11**, 254 (1896) *28*

$CoCl_2$

C_8OH_9NS **III-93**
NH_3

A hydrated cobaltic salt is formed by adding an alcoholic or acetone solution of thioglycolic acid anilide to a neutral solution of a cobaltous salt and then making the mixture ammoniacal. The initial precipitate is red but it forms red-brown flocks on standing. The precipitate after washing and vacuum-drying has the composition, $Co(C_8OH_8NS)_3 \cdot 4H_2O$. The water is lost when the product is warmed in a drying oven at 120° (b).

(a) $$4CoCl_2 + O_2 + 12C_8OH_9NS + 14H_2O + 8NH_3 \rightarrow \underline{4Co(C_8OH_8NS)_3 \cdot 4H_2O} + 8NH_4Cl$$

(b) $$Co(C_8OH_8NS)_3 \cdot 4H_2O \rightarrow Co(C_8OH_8NS)_3 + 4H_2O$$

T. Bersin, Z. anal. Chem., **85**, 430 (1931) *7*

$CoCl_2$

$C_{18}H_{12}N_2$ **III-94**

Cobalt salts form colored coordination compounds with 2,2′-biquinolyl.

$$(CoCl_2 + C_{18}H_{12}N_2 \rightarrow CoCl_2 \cdot C_{18}H_{12}N_2)$$

J. G. Breckenridge, R. W. J. Lewis and L. A. Quinck, Can. J. Res., **B17**, 258 (1939) *69*

$CoCl_2$

H_2O_2 **III-95**

When cobaltous acid is made by the action of hydrogen peroxide on cobaltous chloride, the reaction is possibly an equilibrium reaction.

$$H_2O_2 + CoCl_2 + H_2O \rightarrow H_2CoO_3 + 2HCl$$

A. C. Robertson, J. Am. Chem. Soc. **48**, 2078 (1926) *1*

$CoCl_2$

H_2Se **III-96**

Hydrogen selenide mixed with nitrogen and hydrogen chloride vapor is passed over cobaltous chloride heated to faint red temperature.

$$3CoCl_2 + 4H_2Se \rightarrow Co_3Se_4 + (6HCl + H_2)$$

Fronzes-Diacon, Compt. rend., **131**, 704 (1900) *25*

$CoCl_2$

H_2TiO_3 **III-97**

Cobaltous chloride is fused with titanic acid.

$$CoCl_2 + H_2TiO_3 \rightarrow CoTiO_3 + (2HCl)$$

L. Bourgeois, Bull. Soc. franc. Miner, **15**, 194 (1893) *25*

$CoCl_2$

HgO

$KMnO_4$ **III-98**

A solution of cobaltous chloride is treated with mercuric oxide and potassium permanganate.

$$6CoCl_2 + 5HgO + 2KMnO_4 \rightarrow 5HgCl_2 + 2KCl + 2MnO_2 + 3Co_2O_3$$

C. Winkler, Z. anal. Chem., **3**, 265 (1864) *25*

$CoCl_2$

I_2

Cl_2 **III-99**

Add 15 grams of iodine to a saturated solution of 10 grams of cobaltous chloride, then pass chlorine gas through this solution. Needles of red color, iodine trichloride-cobaltous chloride are obtained. The product crystallizes with $8H_2O$.

$$CoCl_2 + I_2 + 3Cl_2 + 8H_2O \rightarrow \underline{2ICl_3 \cdot CoCl_2 \cdot 8H_2O}$$

R. F. Weinland and F. Schlegelmilch, Z. anorg. Chem., **30**, 134 (1902) *28*

$CoCl_2$

K^+ **III-100**

Cl^-

Changes in color brought about in solutions of cobalt salts by changes in concentration and temperature as well as by addition of certain other halide salts are to be attributed to the formation of complex anions, as shown below.

$$2K^+ + 2Cl^- + xCoCl_2 \rightarrow 2K^+ + Cl_2(CoCl_2)_x^-$$

F. G. Donnan and H. Bassett, J. Chem. Soc. (London), **81**, 939 (1902)
Ref., Kohlschütter, Ber, **37**, 1193 (1904)
Ref., Stewart B. Watkins and Henry G. Denham, J. Chem. Soc. (London), **115**:2, 1269 (1919) *48*

$CoCl_2$

KCN **III-101**

A brownish white precipitate of cobalt cyanide forms when a solution of a cobalt salt reacts with potassium cyanide. This changes to potassium cobaltocyanide on further action with potassium cyanide.

(a) $$(CoCl_2) + 2KCN \rightarrow \underline{Co(CN)_2} + (2KCl)$$

(b) $$Co(CN)_2 + 4KCN \rightarrow K_4Co(CN)_6$$

A. R. Middleton and H. L. Miller, J. Am. Chem. Soc., **38**, 1708 (1916) *1*

$CoCl_2$

KCN **III-102**

Solutions of cobaltous chloride and potassium cyanide are mixed at a low temperature.

$$CoCl_2 + 6KCN \rightarrow K_4Co(CN)_6 + (2KCl)$$

A. Descamps, Ann. chim. phys. [5], **24**, 193 (1881) *25*

$CoCl_2$

KCN **III-103**

Solutions of cobaltous chloride and potassium cyanide are added together.

$$CoCl_2 + 2KCN + 3H_2O \rightarrow \underline{Co(CN)_2 \cdot 3H_2O} + 2KCl$$

Rammelsberg, Pogg. Ann., **42**, 115 (1837)
Ref., Descamps, Ann. chim. phys. [5], **15**, 430 (1878) *25*

$CoCl_2$

KCNO **III-104**

Saturated alcoholic solution of potassium cyanate when added to cobalt salts gives a royal blue color.

$$4KCNO + CoCl_2 \rightarrow K_2Co(CNO)_4 + 2KCl$$

Dorrington and Ward, Analyst., **54**, 327 (1929) *40*

$CoCl_2$

KCNS **III-105**

NH_4CNS

An alcoholic solution of cobaltous chloride will react with an alcoholic solution of potassium thiocyanate yielding potassium cobaltous thiocyanate in solution and potassium chloride in form of a precipitate which is filtered off. The same type of reaction occurs with ammonium thiocyanate.

(a) $$CoCl_2 + 6KCNS \rightarrow K_4Co(CNS)_6 + 2\underline{KCl}$$

(b) $$CoCl_2 + 6NH_4CNS \rightarrow (NH_4)_4Co(CNS)_6 + \underline{2NH_4Cl}$$

P. Walden, Z. anorg. Chem., **23**, 373 (1900) *28*

$CoCl_2$

KHF_2 **III-106**

A mixture of cobaltous chloride and potassium acid fluoride is heated for some hours at 220° and then to the melting temperature. After cooling the mass is extracted with 97% alcohol.

$$CoCl_2 + 2KHF_2 \rightarrow K_2CoF_4 + 2HCl$$

Poulenc, Compt. rend., **114**, 746, (1892) *25*

$CoCl_2$

KHS **III-107**

Potassium hydrogen sulfide reduces cobaltous chloride to metallic cobalt.

$$CoCl_2 + 2KHS \rightarrow Co + (2KCl + H_2S + S)$$

Jacob Myers, Ber., **6**, 440 (1873) *11*

$CoCl_2$

$KMnO_4$ **III-108**

HgO

Potassium permanganate will oxidize cobaltous chloride in the presence of mercuric oxide.

$$6CoCl_2 + 5HgO + 2KMnO_4 + 11H_2O \rightarrow 6Co(OH)_3 + 5HgCl_2 + 2KCl + 2MnO_2 \cdot H_2O$$

C. Winkler, Z. anal. Chem., **3**, 420 (1864)
Ref., J. Am. Chem. Soc., **20**, 174 (1898) *1*

$CoCl_2$

KNO_2 **III-109**

Potassium nitrite is added in excess to a cold solution of cobaltous chloride and the mixture allowed to stand a while.

$$3CoCl_2 + 12KNO_2 + H_2O \rightarrow \underline{3Co(NO_2)_2 \cdot 6KNO_2 \cdot H_2O} + (6KCl)$$

O. L. Erdmann, J. f. prakt. Chem., **97**, 397 (1866)
Sodtler, Am. J. Sci., [2], **49**, 199 (1870) *25*

$CoCl_2$

KNO_2 **III-110**

HNO_2

A solution of cobaltous chloride acidified with acetic acid is mixed with potassium nitrite and nitrous acid.

$$CoCl_2 + 5KNO_2 + 2HNO_2 \rightarrow \underline{K_3Co(NO_2)_6} + 2KCl + NO + H_2O$$

Fischer, Pogg. Ann., **74**, 124 (1848) *25*

$CoCl_2$

KOH **III-111**

CO

In the presence of cysteine, alkaline suspensions of cobalt salts form cobalt carbonyl, when treated with carbon monoxide.

$$2CoCl_2 + 12KOH + 11CO \rightarrow 2KCo(CO)_4 + 3K_2CO_3 + 4KCl + 6H_2O$$

G. W. Coleman and A. A. Blanchard, J. Am. Chem. Soc., **58**, 2160 (1936) *16*

$CoCl_2$

K_2CO_3 **III-112**

Mixtures of concentrated solutions of cobaltous chloride and potassium carbonate are allowed to stand yielding crystals having $4H_2O$.

$$CoCl_2 + 2K_2CO_3 + 4H_2O \rightarrow \underline{K_2CO_3 \cdot CoCO_3 \cdot 4H_2O} + (2KCl)$$

Wood and Jones, Proc. Can. Phil. Soc., **14**, 171 (1908) *25*

$CoCl_2$

$NH_2OH \cdot HCl$ **III-113**

Four moles of hydroxylamine hydrochloride are added to one mole of cobaltous chloride in solution contained in a flask filled with hydrogen and then a few cubic centimeters of alcoholic hydroxylamine added. The mixture is heated on a water bath.

$$CoCl_2 + 2NH_2OH \cdot HCl \rightarrow \underline{CoCl_2 \cdot 2NH_2OH} + (2HCl)$$

W. Feldt, Ber., **27**, 402 (1894) *25*

$CoCl_2$

NH_3 **III-114**

Solid cobaltous chloride absorbs ammonia and swells to a large volume.

$$CoCl_2 + 4NH_3 \rightarrow CoCl_2 \cdot 4NH_3$$

H. Rose, Pogg. Ann., **20**, 157 (1830) *25*

$CoCl_2$

NH_3 **III-115**

Continued passage of ammonia gas through a solution of cobaltous chloride yields a violet precipitate, decomposing to cobaltous chloride diammoniate at 120°.

$$CoCl_2 + 6NH_3 \rightarrow CoCl_2 \cdot 6NH_3$$

A. Naumann, Ber., **37**, 4334 (1904) *25*

$CoCl_2$

NH_3 **III-116**

NO

A hot solution of 17 g. cobalt (II) chloride in 25 g. water is poured into 85 g. concentrated ammonium hydroxide. The solution is then filtered and saturated at room temperature, in the absence of air, with nitric oxide to give a black precipitate of cobalt dichloride mononitroso pentammoniate.

(a) $CoCl_2 + 6NH_3 \rightarrow (CoCl_2 \cdot 6NH_3)$

(b) $CoCl_2 \cdot 6NH_3 + NO \rightarrow \underline{CoCl_2 \cdot NO \cdot 5NH_3} + (NH_3)$

A. Werner and P. Karrer, Helv. Chim., **1**, 60 (1918) *78*

$CoCl_2$

NH_4NO_2 **III-117**

NH_3

When a stream of air is passed through a 20% solution of ammonia in which is dissolved cobalt dichloride and varying amounts of ammonium nitrite, the following reactions take place.

(a) $4CoCl_2 + 8NH_4NO_2 + 12NH_3 + O_2$
$\rightarrow 2H_2O + 4NH_4Cl + \underline{4Co(NO_2)_2Cl \cdot 4NH_3}$

(b) $4CoCl_2 + 4NH_4NO_2 + 16NH_3 + O_2$
$\rightarrow 2H_2O + \underline{4Co(NO_2)Cl_2 \cdot 5NH_3}$

(c) $4CoCl_2 + 12NH_4NO_2 + 8NH_3 + O_2$
$\rightarrow 8NH_4Cl + 2H_2O + \underline{2Co_2(NO_2)_6 \cdot 6NH_3}$

L. M. Jorgensen, Z. anorg. Chem., **7**, 289 (1894) *28*

$CoCl_2$

NH_4OH **III-118**

The precipitation of cobalt hydroxide in aqueous solution is prevented by addition of an excess of ammonia due to the formation of complexes, (b) and (c):

(a) $CoCl_2 + 2NH_4OH \rightarrow Co(OH)_2 + 2NH_4Cl$

(b) $Co(OH)_2 + NH_3 \rightarrow Co(OH)_2 \cdot NH_3$

(c) $CoCl_2 + NH_3 \rightarrow CoCl_2 \cdot NH_3$

M. Chatelet, J. Chim. phys., **33**, 313 (1936) *69*

$CoCl_2$

NH_4OH **III-119**

The precipitation of cobalt hydroxide by ammonium hydroxide, (a), is hindered by ammonium chloride due to formation of ammoniates in solution, (b, c):

(a) $CoCl_2 + 2NH_4OH \rightarrow Co(OH)_2 + 2NH_4Cl$

(b) $CoCl_2 + NH_3 \rightarrow CoCl_2 \cdot NH_3$

(c) $CoCl_2 + 3NH_3 \rightarrow CoCl_2 \cdot 3NH_3$

M. Chatelet, J. Chim. phys., **38**, 23 (1941) *69*

$CoCl_2$

NH_4OH
NO **III-120**

17 Grams of crystalline cobalt chloride are dissolved in 25 cc of water and 85 grams of 25% ammonium hydroxide are added. After several hours, the solution is cooled in ice, and nitric oxide passed into the solution for some time. Black crystals are formed. These are removed by filtration, washed with conc. ammonium hydroxide and dried in vacuo over potassium hydroxide. These crystals are a complex compound, pentamminenitrosocobalt chloride.

$$2CoCl_2 + 10NH_4OH + 2NO \rightarrow Co_2(NO)_2(NH_3)_{10}Cl_4 + 10H_2O$$

J. Sand and O. Genssler, Ann., **329**, 197 (1903) *20*

$CoCl_2$

$(NH_4)_2HPO_4$ **III-121**

A solution of cobaltous chloride is treated with the equivalent amount of ammonium hydrogen orthophosphate, the solution evaporated to dryness, and the residue dried at 100–105°.

$$CoCl_2 + (NH_4)_2HPO_4 + H_2O \rightarrow CoNH_4PO_4 \cdot H_2O + NH_4Cl + HCl$$

Dakin, Z. anal. Chem., **39**, 784 (1900) *25*

$CoCl_2$

$NaHCO_3$ **III-122**

Solutions containing cobaltous chloride and sodium hydrogen carbonate saturated with carbon dioxide are added together and heated to 140° in a sealed tube.

$$CoCl_2 + NaHCO_3 \rightarrow \underline{CoCO_3} + NaCl + HCl$$

Senarmont, Ann. chim. phys. [3], **30**, 129 (1850) *25*

$CoCl_2$

NaOH **III-123**

An excess of sodium hydroxide is added to a cobaltous chloride solution.

$$CoCl_2 + 2NaOH \rightarrow Co(OH)_2 + (2NaCl)$$

Test and Scoles, Proc. Indiana Acad. Sci., **34**, 163, (1924) *25*

$CoCl_2$

$Na_2B_4O_7$ **III-124**

Cobaltous chloride reacts with borax in solution to yield cobalt tetraborate.

$$CoCl_2 + Na_2B_4O_7 \rightarrow CoB_4O_7 + (2NaCl)$$

Rossi, Gazz. chim. ital., **43**, 12, 669 *25*

$CoCl_2$

Na_2CO_3 **III-125**

S

A mixture of one part of cobaltous chloride, 6 parts of sodium carbonate and 6 parts of sulfur is heated for ten minutes in the blast lamp, yielding cobalt trisulfide.

$$2CoCl_2 + 2Na_2CO_3 + 4S \rightarrow Co_2S_3 + (4NaCl + 2CO_2 + SO_2)$$

Fellenberg, Pogg. Ann., **50**, 73 (1849) *25*

$CoCl_2$

Na_2O_2 **III-126**

$H_2C_2O_4$

Cobaltioxalic acid may be obtained in solution by treating a solution of a cobalt salt with a slight excess of sodium peroxide and then treating the cobaltic hydroxide with a cold saturated solution of oxalic acid.

(a) $2CoCl_2 + Na_2O_2 + 4H_2O \rightarrow 2Co(OH)_3 + 2NaCl + 2HCl$

(b) $2Co(OH)_3 + 6H_2C_2O_4 \rightarrow H_6Co_2(C_2O_4)_6 + 6H_2O$

Stanley R. Benedict, J. Am. Chem. Soc., **28**, 172 (1906) *1*

$CoCl_2$

Na_2SO_3 **III-127**

A diluted solution of cobaltous chloride added to a diluted solution of sodium sulfite precipitates basic cobaltous sulfite. The product crystallizes with $10H_2O$.

$$6CoCl_2 + 5Na_2SO_3 + 12H_2O \rightarrow \underline{5CoSO_3 \cdot Co(OH)_2 \cdot 10H_2O} + 10NaCl + 2HCl$$

K. Seubert and M. Elten, Z. anorg. Chem., **4**, 44 (1893) *28*

$CoCl_2$

Na_2SeO_3 **III-128**

When solutions of cobaltous chloride and of sodium selenite are mixed, there precipitates bluish red, amorphous, di-hydrated cobaltous selenite which changes to small prisms when heated.

$$CoCl_2 + Na_2SeO_3 + 2H_2O \rightarrow \underline{CoSeO_3 \cdot 2(H_2O)} + (2NaCl)$$

L. F. Nilson, K. Sven. Vet. Ak. Handl. Övers., **31**, No. 1, 41 (1874) *10*

$CoCl_2$

Na_2TeO_3 **III-129**

Cobalt tellurite is obtained as a dark, purple blue substance, when a solution of sodium tellurite is treated with cobalt chloride.

$$CoCl_2 + Na_2TeO_3 + H_2O \rightarrow \underline{CoTeO_3 \cdot H_2O} + (2NaCl)$$

V. Lenher and E. Wolesensky, J. Am. Chem. Soc., **35**, 728 (1913) *1*

$CoCl_2$

Na_2WO_4 **III-130**

A mixture of two parts of cobaltous chloride, one part of sodium tungstate and two parts of sodium chloride is fused.

$$CoCl_2 + Na_2WO_4 \rightarrow CoWO_4 + 2NaCl$$

H. Schultze, Ann., **126**, 49 (1863)
Ref., Zettnow, Pogg. Ann., **130**, 252 (1867) *25*

$CoCl_2$

PH_3 **III-131**

Phosphine transforms slightly warmed cobaltous chloride into a gray product insoluble in concentrated hydrochloric acid.

$$3CoCl_2 + 2PH_3 \rightarrow Co_3P_2 + (6HCl)$$

H. Rose, Pogg., Ann., **24**, 332 (1832) *25*

$CoCl_2$

ε **III-132**

When a neutral solution of cobalt dichloride is submitted to a process of electrolysis, cobalt is precipitated in the pure state and free chlorine gas is given off.

$$(CoCl_2 \rightarrow \underline{Co} + Cl_2)$$

M. Becquerel, Chem. News, **6**, 126 (1862) *101*

$CoCl_2 \cdot 6H_2O$

Na_2MoO_4 **III-133**

Solutions containing equivalent weights of cobaltous chloride and sodium molybdate are mixed at room temperature and evaporated to crystallization over concentrated sulfuric acid.

$$CoCl_2 \cdot 6H_2O + Na_2MoO_4 \rightarrow \underline{CoMoO_4 \cdot H_2O} + 2NaCl + 5H_2O$$

Marckwald, Dissert. Basel., 1895 *25*

$CoCl_2 \cdot 6H_2O$

$SnCl_4$ **III-134**

Rhombic crystals are obtained when mole quantities of cobalt chloride and stannic chloride in solution are combined and cooled slowly.

$$CoCl_2 \cdot 6H_2O + SnCl_4 \rightarrow CoSnCl_6 \cdot 6H_2O$$

S. M. Jörgensen, Danske, Vid. Selsk. Skr. [5], **6**, (1865) *25*

$CoCl_2 \cdot 6NH_3$

H_2O **III-135**

Cobaltous chloride hexammoniate is decomposed on hydrolysis, forming a precipitate of cobaltous hydroxide and a clear supernatant solution of ammonium chloride.

$$CoCl_2 \cdot 6NH_3 + 2H_2O \rightarrow \underline{Co(OH)_2} + 2NH_4Cl + 4NH_3$$

G. L. Clark, A. J. Quick and W. D. Harkins, J. Am. Chem. Soc., **42**, 2489 (1920) *16*

$CoCl_3$

$C_2H_4N_2S_2$ **III-136**

Cobaltic chloride will react with rubeane yielding an insoluble cobalt salt.

$$2CoCl_3 + 3C_2H_4N_2S_2 \rightarrow \underline{Co_2(C_2H_2N_2S_2)_3} + 6HCl$$

J. V. Dubsky, A. Okac, B. Okac and J. Trtilek, Z. anal. Chem., **98**, 189 (1934) *28*

$CoCl_3$

KNO_2 **III-137**

If cobalt chloride is added to an acidified solution of potassium nitrite, potassium cobaltinitrite is formed, yielding a product crystallizing with $3H_2O$.

$$12KNO_2 + 2CoCl_3 + 3H_2O \rightarrow Co_2(NO_2)_6 \cdot 6KNO_2 \cdot 3H_2O + 6KCl$$

P. C. Rây, J. Chem. Soc. (London), **89:1**, 551 (1906) *103*

$CoCl_3$

$Na_2S_2O_4$ **III-138**

Cobaltic chloride in aqueous solution reacts with sodium dithionite to precipitate cobalt sulfide.

$$2CoCl_3 + 5Na_2S_2O_4 \rightarrow \underline{2CoS} + 6NaCl + 2Na_2SO_4 + 6SO_2$$

O. Brunck, Ann., **336**, 295 (1904) *20*

$CoCl_3(H_2O)\cdot 3NH_3$

NaOH **III-139**

Cobaltichydroxychloride triammoniate is formed by action of sodium hydroxide solution on $CoCl_3(H_2O)\cdot 3NH_3$.

$$2CoCl_3(H_2O)\cdot 3NH_3 + 3NaOH \rightarrow Co_2(OH)_3Cl_3\cdot 6NH_3 + (3NaCl + H_2O)$$

A. Werner, Ber., **40**, 4834 (1907)
Ref., J. J. Sudborough, J. Chem. Soc. (London), **94**:2, 43 (1908) *57*

$CoCl_3(H_2O)\cdot 3NH_3$

Na_2PtCl_6 **III-140**

A saturated solution of hydrated cobaltic chloride triammoniate will react with a concentrated solution of sodium chloroplatinate yielding red crystals.

$$2CoCl_3(NH_3)_3(H_2O) + 3Na_2PtCl_6 + 8H_2O \rightarrow \underline{[Co(NH_3)_3(OH_2)]_2\cdot 3PtCl_6\cdot 8H_2O} + 6NaCl$$

S. M. Jörgensen, Z. anorg. Chem., **5**, 147 (1894) *28*

$CoCl_3\cdot H_2O\cdot 5NH_3$

Decomp. **III-141**

Hydroxocobaltic chloride pentammoniate changes spontaneously into chlorocobaltic chloride pentammoniate.

$$CoCl_3\cdot H_2O\cdot 5NH_3 \rightarrow CoClCl_2\cdot 5NH_3 + H_2O$$

Samuel H. C. Briggs, J. Chem. Soc. (London), **115**:1, 285 (1919) *48*

$CoCl_3 \cdot H_2O \cdot 5NH_3$

K_2CrO_4 **III-142**

If to a solution of hydroxocobaltic chloride pentammoniate (prepared by dissolving 25 grams of the substance in 625 cc of water and 62.5 cc of concentrated ammonia) a solution of potassium chromate (16 grams in 500 cc water) is added, both reactants being kept at 60°, 23 grams of brownish red crystals of chromatocobaltic chloride pentammoniate are obtained. If to the mother liquor (at 50°), more chromate is added, the compound listed under (b) precipitates in the form of yellowish brown prisms.

(a) $CoCl_3 \cdot H_2O \cdot 5NH_3 + K_2CrO_4 \rightarrow \underline{CoCrO_4Cl \cdot 5NH_3} + H_2O + 2KCl$

(b) $(CoCl_3 \cdot H_2O \cdot 5NH_3 + K_2CrO_4 + 4H_2O \rightarrow \underline{CoClCrO_4 \cdot 5H_2O \cdot 5NH_3} + 2KCl)$

Samuel H. C. Briggs, J. Chem. Soc. (London), **115**:1, 70 (1919) *48*

$CoCl_3 \cdot 5NH_3$

AgF **III-143**

A saturated solution of chlorocobaltichloride pentammoniate is treated with a 0.3% solution of silver fluoride. A red precipitate of the fluoride appears and is purified by washing with a 25% solution of ammonium hydroxide.

$$(Co(NH_3)_5Cl)Cl_2 + 2AgF \rightarrow \underline{(Co(NH_3)_5Cl)F_2} + 2AgCl$$

M. S. Litvinoff, J. Gen. Chem. (USSR), **10**, 1491 (1940) *60*

$CoCl_3 \cdot 5NH_3$

$C_2H_8N_2 \cdot H_2O$ **III-144**

The action of hydrated ethylenediamine on cobalt trichloride pentammoniate gives triethylenediaminecobalt trichloride and free ammonia.

$$CoCl_3 \cdot 5NH_3 + 3C_2N_2H_8 \cdot H_2O \rightarrow CoCl_3 \cdot 3C_2N_2H_8 + 5NH_3 + H_2O$$

S. M. Jörgensen, Trans. Faraday Soc., **29**, 626 (1933)
Ref., J. P. Mathieu, J. Chim. phys., **33**, 78 (1936) *69*

$CoCl_3 \cdot 6NH_3$

AgF **III-145**

A saturated solution of cobaltichloride hexammoniate is mixed in the cold with a slight excess of a 0.3% solution of silver fluoride. The precipitated, yellow, crystalline product is freed from silver chloride by washing with 10% ammonium hydroxide.

$$(CoCl_3 \cdot 6NH_3 + 3AgF \rightarrow \underline{CoF_3 \cdot 6NH_3} + 3AgCl)$$

M. S. Litvinoff, J. Gen. Chem. (USSR), **10**, 1490 (1940) *60*

$CoCl_3 \cdot 6(NH_3)$

$K[(NH_3)_2Co(NO_2)_4]$ **III-146**

A solution of luteocobalt chloride will react with a solution of potassium diammino cobalt nitrite yielding a precipitate of orange-yellow color.

$$CoCl_3 \cdot 6(NH_3) + 3K[(NH_3)_2Co(NO_2)_4] \rightarrow \underline{Co(NH_3)_6 \cdot [(NO_2)_2(NH_3)_2Co(NO_2)_2]_3} + 3KCl$$

S. M. Jörgensen, Z. anorg. Chem., **5**, 147 (1894) *28*

$CoCl_3 \cdot 6NH_3$

$Na_3[Co(NO_2)_6]$ **III-147**

A solution of cobaltic chloride hexammoniate will react with a solution of sodium cobaltinitrite yielding a precipitate of luteo cobaltic cobaltinitrite.

$$CoCl_3 \cdot 6NH_3 + Na_3Co(NO_2)_6 \rightarrow \underline{Co_2(NO_2)_6 \cdot 6NH_3} + 3NaCl$$

S. M. Jörgensen, Z. anorg. Chem., **5**, 147 (1894) *28*

$CoCl_3 \cdot 6NH_3$

Δ **III-148**

When cobaltic chloride hexammoniate is heated out of contact with air it decomposes as follows:

$$6(CoCl_3 \cdot 6NH_3) \rightarrow 6CoCl_2 + 6NH_4Cl + N_2 + 28NH_3$$

G. L. Clark, A. J. Quick and W. D. Harkens, J. Am. Chem. Soc., **42**, 2497 (1920) *16*

$CoCrO_4$

HNO_3 **III-149**

Cobalt chromate dissolves in dilute nitric acid.

$$CoCrO_4 + 2HNO_3 \rightarrow Co(NO_3)_2 + H_2CrO_4$$

S. H. C. Briggs, Z. anorg. Chem., **63**, 325 (1909) *25*

$CoCrO_4Cl \cdot 5NH_3$

$AgNO_3$ **III-150**

If silver nitrate be added to a solution of chromato cobaltic chloride pentammoniate, a copious precipitate of silver chloride (colored by traces of silver chromate) is obtained. The mother liquor is reddish yellow.

$$CoCrO_4Cl \cdot 5NH_3 + AgNO_3 \rightarrow CoCrO_4NO_3 \cdot 5NH_3 + \underline{AgCl}$$

Samuel H. C. Briggs, J. Chem. Soc. (London), **115**:1, 70 (1919) *48*

$CoCrO_4Cl \cdot 5NH_3$

Ag_2CrO_4 **III-151**

If freshly prepared silver chromate be poured into a solution of chromato cobaltic chloride pentammoniate and the mixture well shaken, silver chloride will separate. After quickly removing it, glistening scaly crystals of chromato cobaltic chromate decammoniate are formed. The crystals are tri-hydrated.

$$2CoCrO_4Cl \cdot 5NH_3 + Ag_2CrO_4 \rightarrow [CoCrO_4]_2CrO_4 \cdot 10NH_3 + \underline{2AgCl}$$

Samuel H. C. Briggs, J. Chem. Soc. (London), **115**:1, 71 (1919) *48*

$CoCrO_4NO_3 \cdot 5NH_3$

$AgNO_3$ **III-152**

If silver nitrate be added to a cold, freshly prepared solution of chromato cobaltic nitrate pentammoniate, no precipitation will take place. However, upon heating, immediate precipitation of silver chromate and formation of hydroxo cobaltic nitrate pentammoniate will take place. Lead and barium salts behave in exactly the same manner as the silver nitrate.

$$CoCrO_4NO_3 \cdot 5NH_3 + 2AgNO_3 + H_2O$$
$$\rightarrow \underline{Ag_2CrO_4} + Co(NO_3)_3 \cdot H_2O \cdot 5NH_3$$

Samuel H. C. Briggs, J. Chem. Soc. (London), **115**:1, 70 (1919) *48*

CoF_2

$(NH_4)_2F_2$ **III-153**

Cobaltous fluoride is fused with an excess of ammonium fluoride and the residue extracted with boiling alcohol.

$$CoF_2 + (NH_4)_2F_2 \rightarrow CoF_2 \cdot (NH_4)_2F_2$$

Paulenc, Compt. rend., **114**, 1426 (1892) *25*

$CoHPO_3$

Δ **III-154**

Cobalt hydrogen phosphite is ignited.

$$14CoHPO_3 \rightarrow 6Co_2P_2O_7 + 2CoP + 7H_2$$

Rammelsburg, Pogg. Ann., **131**, 372 (1867) *25*

$Co(H_2PO_2)_2$

Δ **III-155**

Cobalt hydrogen hypophosphite is decomposed when heated to 150°.

$$6Co(H_2PO_2)_2 \rightarrow 4Co(PO_3)_2 + 2CoP + 2PH_3 + 9H_2$$

Rammelsburg, Ber. Berl. Akad. (1872), 433 *13*

CoI_2

NH_3 **III-156**

A concentrated cobaltous iodide solution is treated with ammonia yielding a reddish crystalline precipitate.

$$CoI_2 + 4NH_3 \rightarrow \underline{CoI_2 \cdot 4NH_3}$$

Rammelsberg, Pogg. Ann., **48**, 155 (1839) *25*

$CoI_2 \cdot 2H_2O$

H_2O **III-157**

When the green, di-hydrated cobaltous iodide is left standing in the air for some time, it deliquesces to form the red hexa-hydrated cobaltous iodide.

$$CoI_2 \cdot 2H_2O + 4H_2O \rightarrow CoI_2 \cdot 6H_2O$$

Walter N. Hartley, J. Chem. Soc. (London), **27**, 502 (1874) *48*

$CoI_2 \cdot 6H_2O$

Δ **III-158**

Cobalt iodide hexahydrate is completely dehydrated at 130°C leaving the black graphite-like anhydrous salt.

$$CoI_2 \cdot 6H_2O \rightarrow CoI_2 + 6H_2O$$

W. N. Hartley,
Ref., R. Gerstl, Ber , **7**, 740 (1874) *11*

$Co(IO_3)_2$

NH_3 **III-159**

A solution of cobalt (II) iodate in concentrated ammonium hydroxide is saturated with nitric oxide in the absence of air to give a dark brown precipitate of cobalt diiodate mononitrosopentammoniate.

(a) $Co(IO_3)_2 + 6NH_3 \rightarrow (Co(IO_3)_2 \cdot 6NH_3)$

(b) $Co(IO_3)_2 \cdot 6NH_3 + NO \rightarrow \underline{Co(IO_3)_2 \cdot NO \cdot 5NH_3} + (NH_3)$

A. Werner and P. Karrer, Helv. Chim., **1**, 62 (1918) *78*

$Co(NH_2)_2$

NH_3 **III-160**

Cobaltous amide absorbs additional ammonia to form an ammonous cobaltous nitride—analogous to the "hydrous" compounds.

(a) $Co(SCN)_2 + 2KNH_2 \rightarrow 2KSCN + Co(NH_2)_2$

(b) $3Co(NH_2)_2 \rightarrow Co_3N_2 + 4NH_3$ room T

(c) $Co_3N_2 + xNH_3 \rightarrow Co_3N_2 \cdot NH_3$ −40°C

F. W. Bergstrom, J. Phys. Chem., **32**, 433 (1928) *85*

$Co(NH_3)_3(NO_2)_3$

H_2O **III-161**

The dark and light decomposition (λ = 3660 Å) of trinitro triammino cobalt was studied in neutral (a) and acid (b) solutions:

(a) $Co(NH_3)_3(NO_2)_3 + 3H_2O \rightarrow Co(OH)_3 + 3NH_4NO_2$

(b) $2Co(NH_3)_3(NO_2)_3 + H_2O + 4H^+$
$\rightarrow 2Co^{++} + NH_4NO_3 + 5NH_4NO_2$

R. Luther and H. Frieser, Z. Elektrochem., **36**, 141 (1930) *86*

$Co(NH_3)_5Cl_3$

Δ **III-162**

H_2

Na_2CO_3

Cobalt purpureo chloride was heated, forming the chloride. A solution of sodium carbonate was added, forming cobalt carbonate. This was ignited, forming cobalt oxide. This was reduced to cobalt by hydrogen.

(a) $4Co(NH_3)_5Cl_3 + 2H_2O \rightarrow 4CoCl_2 + (16NH_3 + 4NH_4Cl) + O_2$

(b) $CoCl_2 + Na_2CO_3 \rightarrow \underline{CoCO_3} + (2NaCl)$

(c) $CoCO_3 \rightarrow CoO + (CO_2)$

(d) $CoO + H_2 \rightarrow Co + H_2O$

W. A. Tilden, Trans. Roy. Soc. (London), **194**, 236 (1900) *105*

$CoNH_4(SO_3)_2 \cdot 4NH_3$

KOH **III-163**

Potassium hydroxide reacts with cobaltic ammonium sulfite tetrammoniate to yield cobaltic potassium sulfite tetrammoniate, ammonia and water. The corresponding lithium, rubidium and caesium salts are made similarly.

$$CoNH_4(SO_3)_2 \cdot 4NH_3 + KOH \rightarrow CoK(SO_3)_2 \cdot 4NH_3 + NH_3 + H_2O$$

E. H. Riesenfeld, Med. K. Vet. Nobelinst., **6**, No. 6, 9 (1923) *10*

$CoNO(CO)_3$

Br_2 **III-164**

Bromine liberates nitric oxide and carbon monoxide from cobalt nitrosylcarbonyl, (formed by the addition of nitric oxide to an acidified solution of cobalt carbonyl).

$$CoNO(CO)_3 + Br_2 \rightarrow CoBr_2 + NO + 3CO$$

G. W. Coleman and A. A. Blanchard, J. Am. Chem. Soc., **58**, 2160 (1936) *16*

$Co(NO_2)Cl_2 \cdot 4NH_3$

H_2O **III-165**

Monohydrated nitro cobaltic chloride tetrammoniate is formed by dissolving nitrocobaltic chloride tetrammoniate in water.

$$Co(NO_2)Cl_2 \cdot 4NH_3 + H_2O \rightarrow \underline{Co(NO_2)Cl_2(H_2O) \cdot 4NH_3}$$

S. M. Jörgensen, Z. anorg. Chem., **7**, 289 (1894) *28*

$Co(NO_2)Cl_2(H_2O) \cdot 4NH_3$

HBr **III-166**

Red crystals are formed when the water free crystals of hydrated cobalt nitrochloride tetrammoniate react with a solution of hydrobromic acid.

$$Co(NO_2)Cl_2(H_2O) \cdot 4NH_3 + 2HBr \rightarrow \underline{Co(NO_2)Br_2(H_2O) \cdot 4NH_3} + 2HCl$$

S. M. Jörgensen, Z. anorg. Chem., **7**, 289 (1894) *28*

$Co(NO_2)Cl_2(H_2O) \cdot 4NH_3$

HNO_3 **III-167**

Hydrated cobalt nitrochloride tetrammoniate will react with diluted nitric acid yielding yellow colored crystals of hydrated cobalt nitronitrate tetrammoniate.

$$Co(NO_2)Cl_2(H_2O) \cdot 4NH_3 + 2HNO_3 \rightarrow \underline{Co(NO_2)(NO_3)_2(H_2O) \cdot 4NH_3} + 2HCl$$

S. M. Jörgensen, Z. anorg. Chem., **7**, 289 (1894) *28*

$Co(NO_2)Cl_2(H_2O) \cdot 4NH_3$

H_2SO_4 **III-168**

Hydrated cobalt nitro chloride tetrammoniate will react with diluted sulfuric acid yielding yellow colored crystals of hydrated cobalt nitro sulfate tetrammoniate.

$$Co(NO_2)Cl_2(H_2O) \cdot 4NH_3 + H_2SO_4 \rightarrow 2HCl + \underline{Co(NO_2)(SO_4)(H_2O) \cdot 4NH_3}$$

S. M. Jörgensen, Z. anorg. Chem., **7**, 289 (1894) *28*

$Co(NO_2)Cl_2(H_2O)\cdot 4NH_3$

K_2PtCl_4 **III-169**

Hydrated cobalt nitrochloride tetrammoniate will react with a solution of potassium chloroplatinite yielding red colored crystals.

$$Co(NO_2)Cl_2(H_2O)\cdot 4NH_3 + K_2PtCl_4 \rightarrow 2KCl + Co(NO_2)(PtCl_4)(H_2O)\cdot 4NH_3$$

S. M. Jörgensen, Z. anorg. Chem., **7**, 289 (1894) *28*

$Co(NO_2)Cl_2(H_2O)\cdot 4NH_3$

NH_4Cl **III-170**

By heating a solution of monohydrated nitrocobaltic chloride tetrammoniate with a solution of ammonium chloride, the pentammoniate is formed.

$$Co(NO_2)Cl_2(H_2O)\cdot 4NH_3 + NH_4Cl \rightarrow HCl + H_2O + Co(NO_2)Cl_2\cdot 5NH_3$$

S. M. Jörgensen, Z. anorg. Chem., **7**, 289 (1894) *28*

$Co(NO_2)Cl_2(H_2O)\cdot 4NH_3$

NH_4OH **III-171**

The water free crystals of hydrated cobalt nitrochloride tetrammoniate will react with diluted ammonium hydroxide solution yielding red crystals of the basic nitrite.

$$Co(NO_2)Cl_2(H_2O)\cdot 4NH_3 + NH_4OH \rightarrow NH_4Cl + Co(NO_2)Cl(OH)(H_2O)\cdot 4NH_3$$

S. M. Jörgensen, Z. anorg. Chem., **7**, 289 (1894) *28*

$Co(NO_2)Cl_2\cdot H_2O\cdot 4NH_3$

$(NH_4)_2C_2O_4$ **III-172**

Orange red colored crystals are formed when a saturated solution of hydrated cobalt nitro chloride tetrammoniate reacts with a solution of ammonium oxalate.

$$Co(NO_2)Cl_2\cdot 4NH_3\cdot H_2O + (NH_4)_2C_2O_4 \rightarrow 2NH_4Cl + \underline{Co(NO_2)(C_2O_4)(H_2O)\cdot 4NH_3}$$

S. M. Jörgensen, Z. anorg. Chem., **7**, 289 (1894) *28*

$Co(NO_2)Cl_2(H_2O)\cdot 4NH_3$

$NaAuCl_4$ **III-173**

Hydrated cobalt nitro chloride tetrammoniate will react with a solution of sodium chloroaurate yielding crystals of dark red color.

$$Co(NO_2)Cl_2(H_2O)\cdot 4NH_3 + NaAuCl_4 \rightarrow NaCl + Co(NO_2)Cl(AuCl_4)(H_2O)\cdot 4NH_3$$

S. M. Jörgensen, Z. anorg. Chem., **7**, 289 (1894) *28*

$Co(NO_2)Cl_2\cdot 5NH_3$

$NaNO_2$ **III-174**

HNO_3

By heating mononitro cobaltic chloride pentammoniate with a solution of sodium nitrite and nitric acid, croceo cobalt chloride is obtained.

$$Co(NO_2)Cl_2\cdot 5NH_3 + NaNO_2 + HNO_3 \rightarrow NaCl + NH_4NO_3 + Co(NO_2)_2Cl\cdot 4NH_3$$

S. M. Jörgensen, Z. anorg. Chem., **7**, 289 (1894) *28*

$Co(NO_2)(OH)$

HNO_2 **III-175**

If hydroxocobaltonitrite is reacted with nitrous acid, the products are water and cobaltous nitrite.

$$Co(NO_2)(OH) + HNO_2 \rightarrow Co(NO_2)_2 + H_2O$$

T. Suzuki, J. Chem. Soc. (London), **97**:1, 730 (1910) *103*

$Co(NO_2)_2$

NO_2 **III-176**

If cobaltous nitrite is reacted with nitrogen dioxide cobaltic nitrite is formed.

$$Co(NO_2)_2 + NO_2 \rightarrow Co(NO_2)_3$$

T. Suzuki, J. Chem. Soc. (London), **97**:1, 730 (1910) *103*

$Co(NO_2)_2Br \cdot en \cdot pn$

d-3-bromocamphor-7-silver sulfonate **III-177**

Thirty nine g. ethylenediamine propylenediamine cobaltic dinitrite monobromide (the cis salt containing either *d*- or *l*- propylenediamine) is dissolved in two liters of water at 80°C and 47 g. d-3-bromocamphor-7-silver sulfonate added while stirring. Silver bromide precipitates while the ethylenediamine propylenediamine cobaltic dinitrite *d*-3-bromocamphor-7-sulfonate remains in solution

$$Co(NO_2)_2Br \cdot C_2H_4(NH_2)_2 \cdot CH_3CHNH_2CH_2NH_2 + C_{10}H_{14}OBrSO_3Ag \rightarrow \underline{AgBr} + Co(NO_2)_2C_{10}H_{14}OBrSO_3 \cdot C_2H_4(NH_2)_2 \cdot CH_3CHNH_2CH_2NH_2$$

A. Werner, Helv. Chim., **1**, 12, 14 (1918) *78*

$Co(NO_2)_2Br \cdot en \cdot pn$

$(NH_4)_2SO_4$ **III-178**

Ethylenediamine propylenediamine cobaltic dinitrite monobromide (the cis salt containing either *d*- or *l*-propylenediamine) is dissolved in ten times its weight of water and triturated with solid ammonium sulfate. A precipitate of ethylenediamine propylenediamine cobaltic dinitrite hemisulfate is obtained.

$$2Co(NO_2)_2Br \cdot C_2H_4(NH_2)_2 \cdot CH_3CHNH_2CH_2NH_2 + (NH_4)_2SO_4 \rightarrow \underline{Co_2(NO_2)_4SO_4 \cdot 2C_2H_4(NH_2)_2 \cdot 2CH_3CHNH_2CH_2NH_2} + (2NH_4Br)$$

A. Werner, Helv. Chim., **1**, 21 (1918) *78*

$Co(NO_2)_2Br \cdot en \cdot pn$

NaCNS **III-179**

To ethylenediamine propylenediamine cobaltic dinitrite monobromide dissolved in water (the cis salt containing either *d*- or *l*-propylenediamine) is added to an equal weight of sodium thiocyanate. Ethylenediamine propylenediamine cobaltic dinitrite monothiocyanate is obtained as a yellow precipitate.

$$Co(NO_2)_2Br \cdot C_2H_4(NH_2)_2 \cdot CH_3CHNH_2CH_2CN_2 + NaCNS \rightarrow \underline{Co(NO_2)_2CNS \cdot C_2H_4(NH_2)_2 \cdot CH_3CHNH_2CH_2NH_2} + (NaBr)$$

A. Werner, Helv. Chim., **1**, 19 (1918) *78*

$Co(NO_2)_2C_{10}H_{14}OBrSO_3 \cdot en \cdot pn$

HBr **III-180**

Crystals of ethylenediamine propylenediamine cobaltic dinitrite *d*-3-bromocamphor-7-sulfonate (the cis salt containing either *d*- or *l*-propylenediamine) are treated dropwise with conc. hydrobromic acid. Extraction of the resulting paste with absolute alcohol leaves behind crystals of ethylenediamine propylenediamine cobaltic dinitrite monobromide.

$$Co(NO_2)_2C_{10}H_{14}OBrSO_3 \cdot C_2H_4(NH_2)_2 \cdot CH_3CHNH_2CH_2NH_2 + HBr \rightarrow (C_{10}H_{14}OBrSO_3H) + Co(NO_2)_2Br \cdot C_2H_4(NH_2)_2 \cdot CH_3CHNH_2CH_2NH_2$$

A. Werner, Helv. Chim., **1**, 17 (1918) *78*

$Co(NO_2)_2Cl \cdot en \cdot pn$

HCl **III-181**

Gentle warming of ethylenediamine propylenediamine cobaltic dinitrite monochloride (the trans salt containing either *d*- or *l*-propylenediamine) with hydrochloric acid produces ethylenediamine propylenediamine cobaltic mononitrite dichloride.

$$Co(NO_2)_2Cl \cdot C_2H_4(NH_2)_2 \cdot CH_3CHNH_2CH_2NH_2 + HCl \rightarrow CoNO_2Cl_2 \cdot C_2H_4(NH_2)_2 \cdot CH_3CHNH_2CH_2NH_2 + (HNO_2).$$

A. Werner, Helv. Chim., **1**, 31 (1918) *78*

$Co(NO_2)_2Cl \cdot 3NH_3$

HBr **III-182**

H_2SO_4

A solution of dinitrito cobalt chloride triammoniate will react with hydrobromic acid yielding red colored crystals of dinitro cobalt bromide triammoniate. The same type reaction occurs with sulfuric acid, yielding yellow colored crystals of dihydrated tetranitro cobalt sulfate hexammoniate.

(a) $Co(NO_2)_2Cl \cdot 3NH_3 + HBr \rightarrow Co(NO_2)_2Br \cdot 3NH_3 + HCl$

(b) $2Co(NO_2)_2Cl \cdot 3NH_3 + H_2SO_4 + 2H_2O \rightarrow 2HCl + Co_2(NO_2)_4SO_4(H_2O)_2 \cdot 6NH_3$

S. M. Jörgensen, Z. anorg. Chem., **7**, 289 (1894) *28*

$Co(NO_2)_3 \cdot C_2H_4(NH_2)_2 \cdot NH_3$

Propylene diamine **III-183**

Ethylenediamine cobaltic nitrite monammoniate is heated with an equivalent weight of propylenediamine in water solution, stereo isomers of ethylenediamine propylenediamine cobaltic nitrite are formed. Addition of alcohol precipitates the flavo(cis) salts while the croceo (trans) salts remain in solution.

$$Co(NO_2)_3 \cdot C_2H_4(NH_2)_2 \cdot NH_3 + CH_3NH_2CHCH_2NH_2 \rightarrow Co(NO_2)_3 \cdot C_2H_4(NH_2)_2 \cdot CH_3NH_2CHCH_2NH_2 + NH_3$$

A. Werner, Helv. Chim., **1**, 9 (1918) *78*

$Co(NO_2)_3 \cdot en \cdot pn$

HBr **III-184**

Crystals of ethylenediamine propylenediamine cobaltic nitrite (the trans salt containing either *d*- or *l*-propylenediamine) are treated dropwise with cold hydrobromic acid. The product is ethylenediamine propylenediamine cobaltic dinitrite monobromide.

$$Co(NO_2)_3 \cdot C_2H_4(NH_2)_2 \cdot CH_3CHNH_2CH_2NH_2 + HBr \rightarrow Co(NO_2)_2Br \cdot C_2H_4(NH_2)_2 \cdot CH_3CHNH_2CH_2NH_2 + (HNO_2)$$

A. Werner, Helv. Chim., **1**, 32 (1918) *78*

$Co(NO_2)_3 \cdot en \cdot pn$

HCl **III-185**

Crystals of ethylenediamine propylenediamine cobaltic nitrite (the trans salt containing either *d*- or *l*-propylenediamine) are treated dropwise with cold hydrochloric acid. The product is ethylenediamine propylenediamine cobaltic dinitrite monochloride.

$$Co(NO_2)_3 \cdot C_2H_4(NH_2)_2 \cdot CH_3CHNH_2CH_2NH_2 + HCl \rightarrow Co(NO_2)_2Cl \cdot C_2H_4(NH_2)_2 \cdot CH_3CHNH_2CH_2NH_2 + (HNO_2)$$

A. Werner, Helv. Chim., **1**, 31 (1918) *78*

$Co(NO_2)_3 \cdot en \cdot pn$

NaBr **III-186**

Ethylenediamine propylenediamine cobaltic nitrite (the cis salt containing either *d*- or *l*-propylenediamine) is dissolved in three times its weight of water and triturated with one half its weight of solid sodium bromide. Ethylenediamine propylenediamine cobaltic dinitrite monobromide is formed as a precipitate.

$$Co(NO_2)_3 \cdot C_2H_4(NH_2)_2 \cdot CH_3CHNH_2CH_2NH_2 + NaBr \rightarrow \underline{Co(NO_2)_2Br \cdot C_2H_4(NH_2)_2 \cdot CH_3CHNH_2CH_2NH_2} + (NaNO_2)$$

A. Werner, Helv. Chim., **1**, 11 (1918)

78

$Co(NO_2)_3 \cdot 3NH_3$

$C_2H_4(NH_2)_2$ **III-187**

When 10 g. cobaltic nitrite triammoniate is heated with 30 g. of 10% solution of ethylene-diamine and the solution evaporated, crystals of ethylenediamine cobaltic nitrite monoammoniate are obtained.

$$Co(NO_2)_3 \cdot 3NH_3 + C_2H_4(NH_2)_2 \rightarrow Co(NO_2)_3 \cdot C_2H_4(NH_2)_2 \cdot NH_3 + 2NH_3$$

A. Werner, Helv. Chim., **1**, 10 (1918)

78

$Co(NO_2)_3 \cdot 3NH_3$

HCl **III-188**

A solution of cobalt nitrite triammoniate will react with concentrated hydrochloric acid yielding red colored crystals of dinitro cobalt chloride triammoniate.

$$Co(NO_2)_3 \cdot 3NH_3 + HCl \rightarrow Co(NO_2)_2Cl \cdot 3NH_3 + HNO_2$$

S. M. Jörgensen, Z. anorg. Chem., **7**, 289 (1894)

28

$CoNO_3CO_3 \cdot 5NH_3$

HNO_3 **III-189**

If 2.5 grams of cobaltic nitrate carbonate pentammoniate are dissolved in 100 cc of water, and a little dilute nitric acid is added, evolution of carbon dioxide will take place after gently heating the mixture. The main reaction product is hydroxocobaltic nitrate pentammoniate.

$$CoNO_3CO_3 \cdot 5NH_3 + 2HNO_3 \rightarrow Co(NO_3)_3 \cdot H_2O \cdot 5NH_3 + CO_2$$

Samuel H. C. Briggs, J. Chem. Soc. (London), **115**:1, 69 (1919) *48*

$CoNO_3CrO_4 \cdot 4NH_3$

$AgNO_3$ **III-190**

If to a freshly prepared aqueous solution of chromatocobaltic nitrate tetrammoniate a solution of silver nitrate be added and the mixture heated, silver chromate will immediately precipitate, leaving dihydroxocobaltic nitrate tetrammoniate dihydrate, in solution. No immediate reaction takes place after addition of silver nitrate in the cold.

$$CoNO_3CrO_4 \cdot 4NH_3 + 2AgNO_3 + 2H_2O \rightarrow Co(NO_3)_3 \cdot (H_2O)_2 \cdot 4NH_3 + \underline{Ag_2CrO_4}$$

Samuel H. C. Briggs, J. Chem. Soc. (London), **115**:1, 72 (1919) *48*

$CoNO_3CrO_4 \cdot 4NH_3$

$Ba(NO_3)_2$ **III-191**

If to a freshly prepared aqueous solution of chromatocobaltic nitrate tetrammoniate a solution of barium nitrate be added, and the mixture heated, barium chromate will precipitate, leaving diaquo cobaltic nitrate tetrammoniate in solution. The same reaction occurs after letting the mixture stand for several days. No reaction occurs immediately after addition of barium nitrate in the cold.

$$CoNO_3CrO_4 \cdot 4NH_3 + Ba(NO_3)_2 + 2H_2O \rightarrow Co(NO_3)_3 \cdot (H_2O)_2 \cdot 4NH_3 + \underline{BaCrO_4}$$

Samuel H. C. Briggs, J. Chem. Soc. (London), **115**:1, 72 (1919) *48*

$CoNO_3CrO_4 \cdot 4NH_3$

$Pb(NO_3)_2$ **III-192**

If to a freshly prepared aqueous solution of chromatocobaltic nitrate tetrammoniate a solution of lead nitrate be added and the mixture heated, immediate precipitation of lead chromate will take place, leaving hydroxocobaltic nitrate tetrammoniate in solution. The same reaction will take place if the mixture is left standing several days.

$$CoNO_3CrO_4 \cdot 4NH_3 + Pb(NO_3)_2 + 2H_2O \rightarrow Co(NO_3)_3 \cdot (H_2O)_2 \cdot 4NH_3 + \underline{PbCrO_4}$$

Samuel H. C. Briggs, J. Chem. Soc. (London), **115**:1, 72 (1919) *48*

$Co(NO_3)_2$

CSN_2H_4 **III-193**

Blue-green colored crystals are obtained from a cobaltous nitrate solution to which thiourea was added.

$$Co(NO_3)_2 + 4CSN_2H_4 \rightarrow Co(CSN_2H_4)_4(NO_3)_2$$

A. Rosenheim and V. J. Meyer, Z. anorg. Chem., **49**, 13 (1906) *28*

$Co(NO_3)_2$

$C_{13}H_9N$

NH_4CNS **III-194**

Cobaltous nitrate reacts with acridine and ammonium thiocyanate, yielding characteristic crystals.

$$Co(NO_3)_2 + 2C_{13}H_9N + 4NH_4CNS \rightarrow (Co(SCN)_4)H_2(C_{13}H_9N)_2 + (2NH_3 + 2NH_4NO_3)$$

A. Langer, Mikrochem., **25**, 71 (1938) *28*

$Co(NO_3)_2$

$CaOCl_2$ **III-195**

In order to prepare pure cobalt, the nitrate is treated with bleaching powder, giving the oxide, which is dissolved in hydrochloric acid. Then an excess of ammonia is added giving purpureo cobaltchloride; it is heated, and sulfuric acid gives the sulfate, and more ammonia gives the double salt cobalt ammonium sulfate. This is electrolyzed, giving cobalt.

(a) $3CaOCl_2 + 2Co(NO_3)_2 \rightarrow \underline{Co_2O_3} + (2Ca(NO_3)_2 + CaCl_2 + 2Cl_2)$

(b) $Co_2O_3 + 6HCl \rightarrow (2CoCl_3 + 3H_2O)$

(c) $CoCl_3 + 5NH_4OH \rightarrow Co(NH_3)_5Cl_3 + (5H_2O)$

(d) $Co(NH_3)_5Cl_3 \rightarrow CoCl_2 + (4NH_3 + NH_4Cl)$

(e) $CoCl_2 + H_2SO_4 \rightarrow CoSO_4 + (2HCl)$

(f) $CoSO_4 + 2NH_4OH + H_2SO_4 \rightarrow Co(NH_4)_2(SO_4)_2 + 2H_2O$

(g) $2Co(NH_4)_2(SO_4)_4 + (2H_2O) \rightarrow 2Co + (2(NH_4)_2SO_4 + 2H_2SO_4 + O_2)$

W. A. Tilden, Trans. Roy. Soc. (London), **194**, 236 (1900) *105*

$Co(NO_3)_2$

H_2F_2 **III-196**

NH_3

A solution of cobaltous nitrate in hydrofluoric acid is saturated with concentrated ammonia. Upon standing in the air for 48 hours, yellow colored crystals are formed.

$$Co(NO_3)_2 + 24NH_3 + 2H_2F_2 + O_2 \rightarrow Co(NH_3)_6(NO_3)_2F + 2H_2O$$

E. Böhm, Z. anorg. Chem., **43**, 326 (1905) *28*

$Co(NO_3)_2$

$H_4Fe(CN)_6$ **III-197**

Ferrocyanic acid is added to a solution of cobaltous nitrate.

$$2Co(NO_3)_2 + H_4Fe(CN)_6 + 7H_2O \rightarrow Co_2Fe(CN)_6 \cdot 7H_2O + (4HNO_3)$$

Wyrouboff, Ann. chim. phys. [5], **8**, 452 (1876) *25*

$Co(NO_3)_2$

$KHCO_3$ **III-198**

A basic carbonate is precipitated when a 0.05 M. solution of cobalt nitrate is mixed with a 0.5 M. solution of potassium hydrogen carbonate. With more dilute solutions, basic precipitates of various composition are obtained:

$$(5Co(NO_3)_2 + 5KHCO_3 \rightarrow \underline{5CoCO_3 \cdot Co(OH)_2} + 5KNO_3 + 5HNO_3)$$

P. E. Gagnon, L. Cloutier and R. Martineau, Can. J. Res., **B19**, 179 (1941) *69*

$Co(NO_3)_2$

$KHCO_3$ **III-199**

Cobalt nitrate (M/20) was mixed with potassium hydrogen carbonate (M/10) to form a basic carbonate of cobalt.

$$2Co(NO_3)_2 + 4KHCO_3 \rightarrow CoCO_3 \cdot Co(OH)_2 + 4KNO_3 + H_2O + 3CO_2$$

P. E. Gagnon, L. Cloutier and R. Martineau, Can. J. Res., **19B**, 183 (1941) *94*

$Co(NO_3)_2$

KI **III-200**

Cobalt nitrate in strong ammoniacal solution forms a flesh-colored precipitate when potassium iodide is added. Cobaltous iodide hexammoniate is obtained. Chlorides and bromides behave similarly.

$$Co(NO_3)_2 + 6NH_3 + 2KI \rightarrow \underline{CoI_2 \cdot 6NH_3} + 2KNO_3$$

Schoeller and Powell, Analyst, **42**, 189 (1917) *40*

$Co(NO_3)_2$

KIO_3 **III-201**

Solutions of cobalt nitrate and potassium iodate are mixed and heated to 80°–90° in the presence of free nitric acid.

$$Co(NO_3)_2 + 2KIO_3 \rightarrow Co(IO_3)_2 + 2KNO_3$$

A. Ditte, Ann. chim. phys. [6], **21**, 145 (1890) *25*

$Co(NO_3)_2$

K_2CrO_4 **III-202**

Only the normal chromate of cobalt is formed by mixing solutions of cobalt nitrate and of potassium chromate of various concentrations:

$$(Co(NO_3)_2 + K_2CrO_4 \rightarrow \underline{CoCrO_4} + 2KNO_3)$$

P. E. Pelletier, L. Cloutier and P. E. Gagnon, Can. J. Res., **B16**, 37 (1938) *69*

$Co(NO_3)_2$

$K_3Cr(CN)_6$ **III-203**

Cobalt chromicyanide is produced when a 4% solution of cobalt nitrate is added to a 2% solution of potassium chromicyanide.

$$2K_3Cr(CN)_6 + 3Co(NO_3)_2 \rightarrow Co_3Cr_2(CN)_{12} + 6KNO_3$$

F. V. D. Cruser and E. H. Miller, J. Am. Chem. Soc., **28**, 1137 (1906) *1*

$Co(NO_3)_2$

NH_3 **III-204**

NO

A solution of 175 g. cobalt (II) nitrate in 220 g. boiling water is poured into 750 cc. concentrated ammonium hydroxide. After standing 12 hours in the absence of air, nitric oxide is passed into the solution for 2 hours precipitating the red dimer of cobalt dinitrate mononitroso pentammoniate hemihydrate.

(a) $$Co(NO_3)_2 + 6NH_3 \rightarrow (Co(NO_3)_2 \cdot 6NH_3)$$

(b) $$2Co(NO_3)_2 \cdot 6NH_3 + 2NO + H_2O \rightarrow Co_2(NO_3)_4 \cdot 2NO \cdot 10NH_3 \cdot H_2O + (2NH_3)$$

A. Werner and P. Karrer, Helv. Chim. **1**, 62 (1918) *78*

$Co(NO_3)_2$

NH_4OH **III-205**

$C_4O_6H_6$

The neutralization curve of a solution of cobalt nitrate and tartaric acid by ammonia shows the formation of two complex compounds:

(a) $(Co(NO_3)_2 + C_4O_6H_6 + 2NH_4OH \rightarrow CoC_4O_6H_4 + 2NH_4NO_3 + 2H_2O)$

(b) $(Co(NO_3)_2 + C_4O_6H_6 + 3NH_4OH \rightarrow \underline{Co(OH)_2} + NH_4C_4O_6H_5 + 2NH_4NO_3 + H_2O)$

(c) $(Co(NO_3)_2 + C_4O_6H_6 + 4NH_4OH \rightarrow Co(NH_4)_2C_4O_6H_2 + 2NH_4NO_3 + 4H_2O)$

J. L. Delsal, J. Chim. phys., **35**, 314 (1938) *69*

$Co(NO_3)_2$

NH_4VO_3 **III-206**

Ammonium metavanadate reacts with a warm solution of cobaltous nitrate containing free nitric acid.

$$Co(NO_3)_2 + 2NH_4VO_3 + H_2O \rightarrow Co(VO_3)_2 \cdot H_2O + (2NH_4NO_3)$$

A. Ditte, Compt. rend., **104**, 1705 (1887) *25*

$Co(NO_3)_2$

$(NH_4)_2CO_3$ **III-207**

The precipitate obtained by reaction of cobalt nitrate with ammonium carbonate decomposes rapidly on standing:

(a) $(Co(NO_3)_2 + (NH_4)_2CO_3 \rightarrow \underline{CoCO_3} + 2NH_4NO_3)$

(b) $(CoCO_3 + H_2O \rightarrow Co(OH)_2 + CO_2)$

P. E. Gagnon, L. Cloutier and R. Martineau, Can. J. Res., **B19**, 179 (1941) *69*

$Co(NO_3)_2$

$(NH_4)_2CO_3$ **III-208**

Solutions containing cobaltous nitrate and ammonium carbonate in excess are added together and the mixture cooled.

$$Co(NO_3)_2 + 2(NH_4)_2CO_3 + 4H_2O \rightarrow (NH_4)_2CO_3 \cdot CoCO_3 \cdot 4H_2O + (2NH_4NO_3)$$

H. St. Claire Deville, Ann. chim. phys. [3], **35**, 450 (1852) *25*

$Co(NO_3)_2$

NaOH **III-209**

$C_4O_6H_6$

The electrometric titration of solutions of cobalt nitrate and tartaric acid by sodium hydroxide shows the existence of three complex compounds in solution:

(a) $(Co(NO_3)_2 + C_4O_6H_6 + 2NaOH \rightarrow CoC_4O_6H_4 + 2NaNO_3 + 2H_2O)$

(b) $(Co(NO_3)_2 + C_4O_6H_6 + 3NaOH \rightarrow NaCoC_4O_6H_3 + 2NaNO_3 + 3H_2O)$

(c) $(Co(NO_3)_2 + C_4O_6H_6 + 4NaOH \rightarrow Na_2CoC_4O_6H_2 + 2NaNO_3 + 4H_2O)$

J. L. Delsal, J. Chim. phys., **35**, 314 (1938) *69*

$Co(NO_3)_2$

$Na_2H_2Sb_2O_7$ **III-210**

A boiling solution of 3 g. of sodium hydrogen pyroantimonate is treated with a hot concentrated solution of cobaltous nitrate.

$$Co(NO_3)_2 + Na_2H_2Sb_2O_7 + 6H_2O \rightarrow Co(SbO_3)_2 \cdot 7H_2O + (2NaNO_3)$$

A. Ebel, Ber., **22**, 3044 (1889) *25*

$Co(NO_3)_2$

Na_3AsO_3 **III-211**

Cobaltous nitrate is treated with an excess of sodium arsenite.

$$7Co(NO_3)_2 + 4Na_3AsO_3 + H_2O \rightarrow Co_7As_2O_{10} + As_2O_3 + 12NaNO_3 + 2HNO_3$$

C. Reichard, Z. anal. Chem., **42**, 10 (1902) *25*

$Co(NO_3)_2$

O_2 **III-212**

NH_3

When an ammoniacal solution of cobaltous nitrate is oxidized by a slow current of air and an excess of sulfuric acid is added, a complex aminocobaltammoniate is formed.

$$4Co(NO_3)_2 + 18NH_3 + O_2 \rightarrow 2Co_2(OH)(NH_2)(NO_3)_4 \cdot 8NH_3$$

A. Werner, Ber., **40**, 4605 (1907)
Ref., A. McKenzie, J. Chem. Soc. (London), **94:2**, 42 (1908) *57*

$Co(NO_3)_2 \cdot 6NH_3$

K_2HgI_4 **III-213**

Cobaltous nitrate hexammoniate will react with potassium mercury iodide yielding a precipitate.

$$Co(NO_3)_2 \cdot 6NH_3 + 2K_2HgI_4 \rightarrow \underline{Co(HgI_3)_2 \cdot 6NH_3} + 2KI + 2KNO_3$$

A. Taurius, Z. anal. Chem., **101**, 357 (1935) *28*

$Co(NO_3)_3$

$C_9H_{12}ON_2S$ **III-214**

Cobaltic nitrate reacts with 5-cyclohexanone-2-thiohydantoine, yielding a black powder.

$$2Co(NO_3)_3 + C_9H_{12}ON_2S + 5H_2O \rightarrow \underline{C_9H_{10}ON_2S \cdot CoOH \cdot Co(OH)_3 \cdot H_2O} + 6HNO_3$$

I. V. Dubsky, V. Lindelar and V. Cernak, Mikrochem., **25**, 124 (1938) *28*

$Co(NO_3)_3$

$NaNO_2$ **III-215**

$HC_2H_3O_2$

Sodium nitrite is dissolved in an equal weight of hot water. Cobalt nitrate is dissolved in this mixture and glacial acetic acid is then added. After aspirating this solution with air, 95% alcohol is added. Sodium cobaltinitrite is precipitated.

$$Co(NO_3)_3 + 9NaNO_2 + 6HC_2H_3O_2 \rightarrow \underline{Na_3Co(NO_2)_6} + 6NO_2 + (6NaC_2H_3O_2 + 3H_2O)$$

M. Cunningham and F. M. Perkin, J. Chem. Soc. (London), **95:2**, 1568 (1909) *57*

$Co(NO_3)_3 \cdot (H_2O)_2 \cdot 4NH_3$

K_2CrO_4 **III-216**

If to a solution of diaquocobaltic nitrate tetrammoniate, ammonium nitrate is added, and subsequently a solution of potassium chromate dissolved in cold water added drop by drop with vigorous stirring, chromatocobaltic nitrate tetrammoniate is obtained in the form of a dark reddish brown deposit.

$$Co(NO_3)_3 \cdot (H_2O)_2 \cdot 4NH_3 + K_2CrO_4 \rightarrow 2KNO_3 + H_2O + \underline{CoCrO_4NO_3 \cdot H_2O \cdot 4NH_3}$$

Samuel H. C. Briggs, J. Chem. Soc. (London), **115:1**, 72 (1919) *48*

$Co(NO_3)_3 \cdot 6NH_3$

III-217

$Na_3Co(NO_2)_6$

Hexammino cobaltic nitrate (leuteo cobaltic nitrate), when treated with sodium hexanitrito cobaltate, forms cobaltic nitrite triammoniate and sodium nitrate.

$$Co(NH_3)_6(NO_3)_3 + Na_3Co(NO_2)_6 \rightarrow 3NaNO_3 + 2Co(NH_3)_3(NO_2)_3$$

W. Gibbs, Ber., **6**, 833 (1873) *11*

CoO

III-218

Cl_2

CO

A mixture of carbon monoxide and chlorine is passed over heated cobaltous oxide.

$$CoO + CO + Cl_2 \rightarrow CoCl_2 + (CO_2)$$

Quantin, Compt. rend., **104**, 233 (1887) *1*

CoO

III-219

$Co(CN)_2$

Cobaltous oxide oxidizes cobaltous cyanide when heated to a high temperature in an atmosphere of pure dry nitrogen.

(a) $$2CoO + Co(CN)_2 \rightarrow Co_3N_2 + (2CO)$$

(b) $$2CoO + Co(CN)_2 \rightarrow 3Co + (2CO + N_2)$$

Vourinasos, Compt. rend., **168**, 899 (1919) *1*

CoO

III-220

HCl

NH_3

Cobaltous oxide is dissolved in hydrochloric acid and the solution treated with ammonia.

$$CoO + 3HCl + NH_3 + 5H_2O \rightarrow \underline{CoCl_2 \cdot NH_4Cl \cdot 6H_2O}$$

Hautz, Ann., **66**, 284 (1848) *1*

CoO

H_2 **III-221**

Cobaltous oxide is heated in a stream of hydrogen to 500°–600°.

$$CoO + H_2 \rightarrow Co + (H_2O)$$

Glazer, Z. anorg. Chem., **36**, 1 (1903) *1*

CoO

$(HF)_2$ **III-222**

NH_3

A solution of cobaltous oxide in hydrofluoric acid is saturated with concentrated ammonia. On leaving in vacuo for 4 weeks, red colored crystals are obtained.

$$CoO + 2H_2F_2 + 2NH_3 + H_2O \rightarrow \underline{CoF_2 \cdot (NH_4F)_2 \cdot 2H_2O}$$

E. Böhm, Z. anorg. Chem., **43**, 326 (1905) *28*

CoO

H_2F_2 **III-223**

Crystals of acid cobalt fluoride are obtained when a solution of cobaltous oxide in hydrofluoric acid is left in vacuo over sulfuric acid.

$$CoO + 7HF + 5H_2O \rightarrow \underline{CoF_2(HF)_5 \cdot 6H_2O}$$

E. Böhm, Z. anorg. Chem., **43**, 326 (1905) *28*

CoO

H_2O_2 **III-224**

When cobalt monoxide is oxidized in the presence of an alkali hydroxide, the alkali hydroxide prevents the formation of the higher oxides of cobalt so cobaltic hydrate is formed.

$$2CoO + H_2O_2 + 2H_2O \rightarrow Co_2O_3 \cdot 3H_2O$$

A. H. McConnell and E. S. Hanes, J. Chem. Soc. (London), **71**:1, 589 (1897) *103*

CoO

III-225

H_2O_2

Cobaltous oxide is oxidized to cobalt dioxide with hydrogen peroxide. The cobalt dioxide dissolves in the water formed (or present) to yield cobaltous acid.

(a) $$CoO + H_2O_2 \rightarrow CoO_2 + H_2O$$

(b) $$CoO_2 + H_2O \rightarrow H_2CoO_3$$

A. H. McConnell and E. S. Hanes, J. Chem. Soc. (London), **71**:1, 586 (1897) *103*

CoO

III-226

H_2S

When black cobalt monoxide is heated to redness in the presence of hydrogen sulfide, it is converted to the yellow cobalt monosulfide.

$$(CoO + H_2S \rightarrow CoS + H_2O)$$

Th. Hjortdahl, Chem. News, **2**, 47 (1868) *101*

CoO

III-227

H_2SO_4

Cobaltous oxide is dissolved in dilute sulfuric acid and the solution concentrated.

$$CoO + H_2SO_4 + 6H_2O \rightarrow \underline{CoSO_4 \cdot 7H_2O}$$

Oechanerde Coninck, Bull. Acad. (Belg.), 833 (1904) *1*

CoO

III-228

NH_4OH

H_2SO_4

I

Iodine added to an ammoniacal solution of cobaltous oxide in sulfuric acid precipitates amorphous, yellow luteo cobaltous amminio iodide sulfate which later changes to small, yellow, octahedral crystals.

$$2CoO + 2H_2SO_4 + 12NH_4OH + I_2 \rightarrow \underline{Co_2(NH_3)_{12} \cdot (SO_4)_2 \cdot I_2} + 14H_2O$$

J. M. Krok, Acta Univ. Lund., **78**, No. 5, 3 (1871) *10*

CoO

$Pr(NO_3)_3$ **III-229**

A soluble double salt is formed when a solution of a rare earth salt (nitrate etc.) is treated with nickel, cobalt or ferrous oxides or carbonates.

$$3CoO + 4Pr(NO_3)_3 + 24H_2O \rightarrow 3Co(NO_3)_2 \cdot 2Pr(NO_3)_3 \cdot 24H_2O + \underline{(Pr_2O_3)}$$

A. C. Neish and J. W. Burns, Can. Chem. & Met., **5**, 72 (1921) *15*

CoO

S **III-230**

A mixture of 1 part of cobaltous oxide and three parts of sulfur is heated almost to glowing in a glass retort.

$$2CoO + 5S \rightarrow 2CoS_2 + SO_2$$

Setterberg, Pogg. Ann., **7**, 40 (1826) *1*

CoO

SiO_2 **III-231**

An intimate mixture of cobaltous oxide and cobaltous chloride is heated for several hours to bright redness with an excess of amorphous silicon dioxide.

$$2CoO + SiO_2 \rightarrow Co_2SiO_4$$

Bourgeois, Compt. rend., **108**, 1177 (1889) *1*

CoO_2

H_2O_2 **III-232**

When cobalt dioxide is treated with hydrogen peroxide, cobaltous acid is formed and oxygen set free.

$$(2CoO_2 + 2H_2O_2 \rightarrow 2H_2CoO_3 + O_2)$$

A. H. McConnell and E. S. Hanes, J. Chem. Soc. (London), **71:1**, 587 (1897) *103*

$Co(OH)_2$

BaO_2 **III-233**

Cobaltous hydroxide is oxidized in the cold by treating with barium peroxide.

$$2Co(OH)_2 + BaO_2 \rightarrow Co_2O_3 + BaO + 2H_2O$$

C. Reichard, Z. anal. Chem., **42**, 10 (1903) *25*

$Co(OH)_2$

$HClO_4$ **III-234**

Cobaltous hydroxide is treated with perchloric acid.

$$Co(OH)_2 + 2HClO_4 \rightarrow Co(ClO_4)_2 + 2H_2O$$

Goldblum and Terlikowski, Bull. Soc. Chim. [IV], **11**, 103 (1912) *26*

$Co(OH)_2$

$H_2C_3H_2O_4$ **III-235**

Dihydrated cobaltous malonate may be prepared by neutralizing malonic acid with a slurry of cobaltous hydroxide. The compound, which has a deep red color, is deposited by evaporating the solution on a water bath. Dihydrated cobalt hydrogen malonate is prepared in the same manner as the normal salt except that an excess of acid is used. This compound has a pinkish red color.

(a) $$Co(OH)_2 + H_2C_3H_2O_4 \rightarrow CoC_3H_2O_4 \cdot 2H_2O$$

(b) $$Co(OH)_2 + 2H_2C_3H_2O_4 \rightarrow CoH_2(C_3H_2O_4)_2 \cdot 2H_2O$$

R. C. Lord, J. Phys. Chem., **11**, 173 (1907) *7*

$Co(OH)_2$

H_2F_2 **III-236**

Cobalt fluoride is formed when cobaltous hydroxide is treated with hydrofluoric acid.

$$Co(OH)_2 + H_2F_2 \rightarrow CoF_2 + 2H_2O$$

F. H. Edmister and H. C. Cooper, Science, **50**, 444 (1919)
Ref., J. Am. Chem. Soc., **42**, 2423 (1920) *1*

$Co(OH)_2$

H_2F_2 **III-237**

Large garnet-red crystals are obtained when freshly prepared cobalt hydroxide is dissolved in hydrofluoric acid.

$$Co(OH)_2 + H_2F_2 + 2H_2O \rightarrow CoF_2 \cdot 4H_2O$$

Costachescu, Ann. Scient. Univ. Jassy, **7**, 5
Ref., J. L. Howe, J. Am. Chem. Soc., **34**, 154 (1912) *1*

$Co(OH)_2$

H_2O_2 **III-238**

Cobaltous acid is formed when hydrogen peroxide acts upon cobaltous hydroxide.

$$Co(OH)_2 + H_2O_2 \rightarrow H_2CoO_3 + (H_2O)$$

A. H. McConnell and E. S. Hanes, J. Chem. Soc. (London), **71**, 584 (1897)
Ref., A. C. Robertson, J. Am. Chem. Soc., **48**, 2077 (1926) *1*

$Co(OH)_2$

$H_2Pt(C_2O_4)_2$ **III-239**

Cobaltous hydroxide (or carbonate) treated with platino-oxalic acid yields fine, copper-colored needles of octahydrated cobaltous platino-oxalate.

$$Co(OH)_2 + H_2Pt(C_2O_4)_2 + 6H_2O \rightarrow CoC_2O_4 \cdot PtC_2O_4 \cdot 8H_2O$$

H. G. Söderbaum, K. Sven. Vet. Ak. Handl. Övers., **42**, No. 10, 34 (1885) *10*

$Co(OH)_2$

H_3PO_2 **III-240**

A solution of freshly precipitated hypophosphorous acid is treated with cobaltous hydroxide in the cold and the solution evaporated in a vacuum.

$$Co(OH)_2 + 2H_3PO_2 + 4H_2O \rightarrow Co(H_2PO_2)_2 \cdot 6H_2O$$

H. Rose, Pogg. Ann., **12**, 87 (1828) *25*

$Co(OH)_2$

KIO_3 **III-241**

Cobaltous hydroxide is oxidized by treating with an excess of potassium iodate.

$$6Co(OH)_2 + KIO_3 + 3H_2O \rightarrow 6Co(OH)_3 + KI$$

Moody, Am. J. Sci. [4], **22**, 176 (1906) *25*

$Co(OH)_2$

KOH **III-242**

Cobaltous hydroxide reacts with potassium hydroxide.

$$Co(OH)_2 + 2KOH \rightarrow K_2CoO_2 + 2H_2O$$

Winkler, J. f. prakt. Chem., **91**, 358 (1864) *25*

$Co(OH)_2$

$(NH_4)_2F_2$ **III-243**

Cobaltous hydroxide will dissolve in a hot solution of ammonium fluoride yielding red crystals after the excess of water has been evaporated.

$$Co(OH)_2 + 2(NH_4)_2F_2 \rightarrow \underline{CoF_2 \cdot (NH_4)_2F_2 \cdot 2H_2O} + 2NH_3$$

H. V. Helmholt, Z. anorg. Chem., **3**, 115 (1893) *28*

$Co(OH)_2$

$(NH_4)_2MoO_4$ **III-244**

Freshly precipitated cobaltous hydroxide is digested with a solution of ammonium molybdate containing free ammonia and the solution evaporated.

$$Co(OH)_2 + (NH_4)_2MoO_4 \rightarrow \underline{CoMoO_4 \cdot 2NH_3 \cdot H_2O} + H_2O$$

Sonnenschein, J. f. prakt. Chem., **53**, 340 (1851) *25*

$Co(OH)_2$

Tl_2O **III-245**

HCN

A mixture of cobaltous hydroxide, hydrocyanic acid and thallous oxide yields thallous cobalticyanide.

$$2Co(OH)_2 + 3Tl_2O + 12HCN \rightarrow 2Tl_3Co(CN)_6 + (7H_2O + H_2)$$

C. Fronmüller, Ber., **11**, 91 (1878) *25*

$Co(OH)_3$

KI **III-246**

HCl

Cobaltic hydroxide, when treated with acids and potassium iodide, liberates iodine and produces a cobaltous salt.

$$2Co(OH)_3 + 2KI + 6HCl \rightarrow 2CoCl_2 + I_2 + 2KCl + 6H_2O$$

W. D. Engle and R. G. Gustavson, J. Ind. Eng. Chem., **8**, 901 (1916) *22*

$Co(OH)_3$

KI **III-247**

H_2SO_4

Cobaltic hydroxide is reduced by potassium iodide in the presence of sulfuric acid.

$$2Co(OH)_3 + 2KI + 3H_2SO_4 \rightarrow 2CoSO_4 + I_2 + 6H_2O + K_2SO_4$$

W. D. Engle and R. G. Gustavson, J. Ind. Eng. Chem., **8**, 901 (1916)
Ref., Willard and Hall, J. Am. Chem. Soc., **44**, 2238 (1922) *1*

$Co(OH)_3$

$SnCl_2$ **III-248**

HCl

For volumetrically determining cobalt, reduction by standardized stannous chloride in conc. HCl and titration of excess $SnCl_2$ with any of several oxidizing agents is satisfactory.

$$2Co(OH)_3 + SnCl_2 + 6HCl \rightarrow 2CoCl_2 + SnCl_4 + 6H_2O$$

H. H. Willard and D. Hall, J. Am. Chem. Soc., **44**, 2238 (1922) *1*

$Co(OH)_3$

$Ti_2(SO_4)_3$ **III-249**

Cobaltic hydroxide is reduced by titanous sulfate, excess of which may be titrated with permanganate for a volumetric determination of cobalt.

$$2Co(OH)_3 + Ti_2(SO_4)_3 + 3H_2SO_4 \rightarrow 2Ti(SO_4)_2 + 2CoSO_4 + 6H_2O$$

H. H. Willard and D. Hall, J. Am. Chem. Soc., **44**, 2246 (1922) *1*
16

CoS

$Pb(NO_3)_2$ **III-250**

Cobalt sulfide is added to a solution of lead nitrate.

$$CoS + Pb(NO_3)_2 \rightarrow Co(NO_3)_2 + \underline{PbS}$$

E. Schurmann, Ann., **249**, 326 (1888) *25*

$CoSO_4$

$Ba(BrO_3)_2$ **III-251**

A solution of cobaltous sulfate is treated with barium bromate. The solution is filtered and evaporated.

$$CoSO_4 + Ba(BrO_3)_2 + 6H_2O \rightarrow \underline{BaSO_4} + Co(BrO_3)_2 \cdot 6H_2O$$

Rammelsburg, Pogg. Ann., **55**, 71, (1842) *25*

$CoSO_4$

$Ba(C_2H_5SO_4)_2$ **III-252**

Solutions of cobalt sulfate and barium ethyl sulfate are mixed, the barium sulfate filtered off, and the filtrate yields large, especially pretty, well-formed, brownish red, stable crystals of hexa-hydrated cobalt ethyl sulfate.

$$CoSO_4 + Ba(C_2H_5SO_4)_2 + 6H_2O \rightarrow \underline{BaSO_4} + Co(C_2H_5SO_4)_2 \cdot 6H_2O$$

J. E. Alèn, K. Sven. Vet. Ak. Handl. Övers., **37**, No. 8, 37 (1880) *10*

$CoSO_4$

$Ba(CNS)_2$ **III-253**

Solutions containing equimolecular quantities of cobaltous sulfate and barium thiocyanate are combined and evaporated after filtration.

$$CoSO_4 + Ba(CNS)_2 + 4H_2O \rightarrow \underline{BaSO_4} + Co(CNS)_2 \cdot 4H_2O$$

H. Grossmann and F. Hunseller, Z. anorg. Chem., **46**, 368 (1905). *25*

$CoSO_4$

$BaCO_3$ **III-254**

HCN

A solution containing one mole of cobaltous sulfate is treated with two moles of barium carbonate and an excess of hydrocyanic acid. The mixture is filtered and evaporated.

$$2CoSO_4 + 4BaCO_3 + 12HCN$$
$$\rightarrow Ba_3(Co(CN)_6)_2 + (\underline{BaSO_4} + H_2SO_4 + 4H_2O + 4CO_2 + H_2)$$

P. Weselsky, Ber., **2**, 590 (1869) *25*

$CoSO_4$

$Ba(ClO_3)_2$ **III-255**

A cobaltous sulfate solution is treated with barium chlorate, filtered, and the filtrate evaporated over sulfuric acid below 18°C.

$$CoSO_4 + Ba(ClO_3)_2 + 6H_2O \rightarrow \underline{BaSO_4} + Co(ClO_3)_2 \cdot 6H_2O$$

Wachter, J. f. prakt. Chem., **30**, 371 (1834)
Ref., A. Meusser, Ber., **35**, 1417 (1902) *25*

$CoSO_4$

$Ba(NO_2)_2$ **III-256**

BeF_2

From a solution of cobalt sulfate, barium nitrite precipitates barium sulfate and leaves cobalt nitrite in solution. Enough ammonium fluoride in solution was added to react with the cobalt nitrite and the solution evaporated and heated to decomposition, leaving cobalt fluoride. This was treated with beryllium fluoride to form cobalt fluoberyllate. Equimolecular amounts of cobalt fluoberyllate and ammonium fluoberyllate were mixed and allowed to crystallize to form cobalt ammonium fluoberyllate.

(a) $CoSO_4 + Ba(NO_2)_2 \rightarrow \underline{BaSO_4} + Co(NO_2)_2$

(b) $Co(NO_2)_2 + 2NH_4F \rightarrow CoF_2 + 2N_2 + 4H_2O$

(c) $CoF_2 + BeF_2 \rightarrow CoBeF_4$

(d) $CoBeF_4 + (NH_4)_2BeF_4 + 6H_2O \rightarrow CoBeF_4 \cdot (NH_4)_2BeF_4 \cdot 6H_2O$

K. S. Krishman and A. Mookherji, Trans. Roy. Soc. (London), **237A**, 136 (1939) *105*

$CoSO_4$

BaS_2O_3 **III-257**

$(NH_4)_2S_2O_3$

A solution of cobalt sulfate is shaken with finely dispersed barium thiosulfate for several days. To this solution containing cobalt thiosulfate an excess of ammonium thiosulfate is added. By evaporating in vacuo over sulfuric acid, greenish blue colored prisms of ammonium cobalto thiosulfate are formed.

(a) $CoSO_4 + BaS_2O_3 \rightarrow CoS_2O_3 + \underline{BaSO_4}$

(b) $2CoS_2O_3 + 3(NH_4)_2S_2O_3 + 6H_2O \rightarrow \underline{(NH_4)_6Co_2(S_2O_3)_5 \cdot 6H_2O}$

A. Rosenheim and I. Davidsohn, Z. anorg. Chem., **41**, 231 (1904) *28*

$CoSO_4$

BaS_2O_6 **III-258**

A solution of cobaltous sulfate is treated with barium dithionate, filtered, and the filtrate evaporated at a low temperature.

$$CoSO_4 + BaS_2O_6 \rightarrow CoS_2O_6 + \underline{BaSO_4}$$

Heeren, Pogg. Ann., **7**, 55 (1826) *25*

$CoSO_4$

C **III-259**

Cobaltous sulfate is heated in the electric furnace with carbon.

$$CoSO_4 + 4C \rightarrow CoS + 4CO$$

A. Mourlot, Ann. chim. phys. [7], **17**, 546 (1899) *25*

$CoSO_4$

CSN_2H_4 **III-260**

Thiourea, finely powdered, is added to a boiling solution of cobaltous sulfate. A dark blue colored crystalline powder is obtained.

$$2CoSO_4 + 3CSN_2H_4 \rightarrow Co_2(CSN_2H_4)_3(SO_4)_2$$

A. Rosenheim and V. J. Meyer, Z. anorg. Chem., **49**, 13 (1906) *28*

$CoSO_4$

$C_7H_6O_2NNa$ **III-261**

A solution of cobalt sulfate will react with the sodium salt of anthranilic acid yielding a crystalline red precipitate.

$$CoSO_4 + 2C_7H_6O_2NNa \rightarrow \underline{Co(C_7H_6O_2N)_2} + Na_2SO_4$$

H. Funk and M. Ditt, Z. anal. Chem., **93**, 241 (1933) *28*

$CoSO_4$

$CaCO_3$ **III-262**

NO

NO_2

Basic cobalto-cobaltic nitrite is prepared by adding cobaltous sulfate to precipitated calcium carbonate suspended in water and dissolved by addition of nitrous gases.

(a) $$CaCO_3 + NO + NO_2 \rightarrow Ca(NO_2)_2 + CO_2$$

(b) $$20Ca(NO_2)_2 + 20CoSO_4 + 44H_2O \rightarrow \underline{20CaSO_4} + 6NO + 4HNO_2 + 6Co^{++}14Co^{+++}(NO_2)_{30}(OH)_{16}\cdot 34H_2O$$

T. Suzuki, J. Chem. Soc. (London), **97**, 727 (1910) *57*

$CoSO_4$

H_2O **III-263**

The author states that in solutions of cobalt sulfate of molar volume 4 to 32, the following ionic reaction takes place to the extent of 0.0149% at 25°.

$$Co^{++} + 2(OH^-) \rightarrow Co(OH)_2$$

H. G. Denham, J. Chem. Soc. (London), **93**, 61 (1908) *57*

$CoSO_4$

H_2S **III-264**

By passing hydrogen sulfide through a solution of cobaltous sulfate to which tartaric acid and sodium carbonate have been added, the cobalt is precipitated quantitatively.

$$CoSO_4 + H_2S \rightarrow \underline{CoS} + H_2SO_4$$

Villiero, Compt. rend., **119**, 1263 (1894) *25*

$CoSO_4$

H_3PO_4 **III-265**

Cobaltous sulfate is added to an excess of orthophosphoric acid and the solution evaporated to dryness and the residue heated to 316°.

$$CoSO_4 + 2H_3PO_4 \rightarrow Co(PO_3)_2 + 3H_2O + SO_3$$

R. Maddrell, Ann., **61**, 53 (1847) *25*

$CoSO_4$

KCN **III-266**

A reddish brown precipitate is formed when potassium cyanide is added in excess to a solution of cobaltous sulfate.

$$CoSO_4 + 4KCN \rightarrow K_2Co(CN)_4 + (K_2SO_4)$$

T. Tatersall, Chem. News, **39**, 66 (1879) *25*

$CoSO_4$

KCNS **III-267**

NH_4CNS

A solution of cobaltous sulfate will react with a solution of potassium thiocyanate yielding potassium cobaltous thiocyanate and potassium sulfate. The potassium sulfate is eliminated by the addition of alcohol. The same type reaction occurs with ammonium thiocyanate.

(a) $CoSO_4 + 6KCNS \rightarrow K_4Co(CNS)_6 + K_2SO_4$

(b) $CoSO_4 + 6(NH_4)CNS \rightarrow (NH_4)_4Co(CNS)_6 + (NH_4)_2SO_4$

P. Walden, Z. anorg. Chem., **23**, 373 (1900) *28*

$CoSO_4$

KIO_3 **III-268**

KI

Cobaltic hydroxide is formed by the reaction of cobaltous sulfate with a mixture of potassium iodate and iodide.

(a) $3CoSO_4 + 5KI + KIO_3 + 3H_2O \rightarrow 3Co(OH)_2 + 3K_2SO_4 + 6I$

(b) $6Co(OH)_2 + KIO_3 + 3H_2O \rightarrow 6Co(OH)_3 + KI$

S. E. Moody, Z. anorg. Chem., **51**, 121 (1906) *28*

Ref., St. R. Benedict, Am. Chem. J., **34**, 581 (1905) *25*

$CoSO_4$

$KMnO_4$ **III-269**

H_2O_2

To a cooled mixture of 7 g. cobaltous sulfate and 4 g. of potassium permanganate in 200 cc. of water, hydrogen peroxide is added slowly with stirring.

$$2CoSO_4 + 2KMnO_4 + 5H_2O_2 \rightarrow \underline{2CoMnO_3 \cdot 2H_2O} + (K_2SO_4 + H_2SO_4 + 4O_2)$$

M. Salinger, Z. anorg. Chem., **33**, 351 (1903) *25*

$CoSO_4$

KNO_2 **III-270**

NH_3

When to a solution of cobalt sulfate is added potassium nitrite and ammonia, and allowed to stand for several days, yellow crystals of dinitrito tetra ammino cobaltic sulfate appear. The solution takes up oxygen from the air.

$$2CoSO_4 + 8NH_3 + 4KNO_2 + O + H_2O \rightarrow K_2SO_4 + Co_2(NH_3)_8(NO_2)_4SO_4 + 2KOH$$

W. Gibbs, Ber., **6**, 831 (1873) *11*

$CoSO_4$

KOH **III-271**

Cobaltous sulfate reacts with potassium hydroxide.

$$CoSO_4 + 4KOH \rightarrow K_2CoO_2 + K_2SO_4 + 2H_2O$$

Winkler, J. f. prakt. Chem., **91**, 358 (1864) *25*

$CoSO_4$

KOH **III-272**

$K_2S_2O_8$

Cobaltous sulfate will react with potassium hydroxide yielding cobaltous hydroxide. By heating this precipitate of cobaltous hydroxide for several hours on a water bath with potassium persulfate, cobaltic hydroxide, a precipitate of brown-black color is obtained.

(a) $$CoSO_4 + 2KOH \rightarrow \underline{Co(OH)_2} + K_2SO_4$$

(b) $$2Co(OH)_2 + K_2S_2O_8 + 2H_2O \rightarrow \underline{2Co(OH)_3} + K_2SO_4 + H_2SO_4$$

E. Hüttner, Z. anorg. Chem., **27**, 81 (1901) *28*

$CoSO_4$

K_2SeO_4 **III-273**

Aqueous solutions of cobalt sulfate and potassium selenate were mixed in equimolecular proportions and allowed to crystallize, forming the double salt cobalt potassium sulfato-selenate. Nickel ammonium sulfato-selenate was formed in a similar method.

$$CoSO_4 + K_2SeO_4 + 6H_2O \rightarrow CoSO_4 \cdot K_2SeO_4 \cdot 6H_2O$$

K. S. Krishman and A. Mookherji, Trans. Roy. Soc. (London), **237A**, 136 (1939) *105*

$CoSO_4$

$K_4Fe(CN)_6$ **III-274**

Potassium ferrocyanide is added to an ammoniacal solution of cobaltous sulfate.

$$2CoSO_4 + K_4Fe(CN)_6 \rightarrow Co_2Fe(CN)_6 + 2K_2SO_4$$

Grossamann, Chem. Ztg., **31**, 37 (1907) *25*

$CoSO_4$

NH_3 **III-275**

Dry cobaltous sulfate absorbs about 66% of ammonia with swelling and evolution of heat.

$$CoSO_4 + 6NH_3 \rightarrow CoSO_4 \cdot 6NH_3$$

H. Rose, Pogg. Ann., **20**, 152 (1830) *25*

$CoSO_4$

NH_3

NO **III-276**

100 g. cobalt (II) sulfate dissolved in 125 g. boiling water is poured into 450 g. concentrated ammonium hydroxide. After standing 12 hours the solution is saturated with nitrogen oxide to precipitate the red dimer of cobalt monosulfate mononitroso pentammoniate monohydrate.

(a) $$CoSO_4 + 6NH_3 \rightarrow (CoSO_4 \cdot 6NH_3)$$

(b) $$2CoSO_4 \cdot 6NH_3 + 2NO + 2H_2O \rightarrow \underline{Co_2(SO_4)_2 \cdot 2NO \cdot 10NH_3 \cdot 2H_2O} + (2NH_3)$$

A. Werner and P. Karrer, Helv. Chim., **1**, 68 (1918) *78*

$CoSO_4$

NH_4CNS **III-277**

Cobaltous sulfate, in solution, is treated with ammonium thiocyanate and the resulting solution extracted with amyl alcohol and the extract evaporated over concentrated sulfuric acid.

$$CoSO_4 + 4NH_4CNS \rightarrow (NH_4)_2Co(CNS)_4 + (NH_4)_2SO_4$$

E. P. Treadwell, Z. anorg. Chem., **26**, 109 (1901) *26*

$CoSO_4$

NH_4OH **III-278**

I_2

When iodine is added to an ammoniacal solution of cobaltous sulfate and the resulting filtrate evaporated spontaneously very small, octahedral crystals of dihydrated rodeo cobaltous ammonia sulfate iodide are formed.

$$2CoSO_4 + 10NH_4OH + I_2 \rightarrow Co_2(NH_3)_{10}\cdot(SO_4)_2I_2\cdot 2(H_2O) + (8H_2O)$$

J. M. Krok, Acta Univ. Lund., **78**, No. 5, 15 (1871) *10*

$CoSO_4$

$(NH_4)_2SO_4$ **III-279**

Equimolecular quantities of cobaltous sulfate and ammonium sulfate in aqueous solution were mixed and allowed to crystallize in order to form ammonium cobaltous sulfate hexahydrate.

$$(NH_4)_2SO_4 + CoSO_4 + 6H_2O \rightarrow (NH_4)_2Co(SO_4)_2\cdot 6H_2O$$

A. E. H. Tutton, Trans. Roy. Soc. (London), **216A**, 13 (1916) *105*

$CoSO_4$

$(NH_4)_3AsO_4$ **III-280**

Red microscopic needles are obtained when solutions of cobaltous sulfate and ammonium arsenate are combined (free ammonia being absent).

$$3CoSO_4 + 2(NH_4)_3AsO_4 + 8H_2O \rightarrow Co_3(AsO_4)_2\cdot 8H_2O + (3(NH_4)_2SO_4)$$

O. Ducru, Ann. chim. phys. [7] **22**, 190 (1901) *25*

$CoSO_4$

NO

NH_4OH

III-281

20 g of crystalline cobalt sulfate are dissolved in 25 cc of boiling water and 85 g of conc. ammonium hydroxide added. The solution is allowed to stand five hours and it is then saturated with nitric oxide. The color of the solution changes to a wine red from which a dark red crystalline powder separates. This is a mixture of the 2 compounds listed below.

$$6CoSO_4 + 29NH_4OH + 6NO \rightarrow Co_2(N_2O_2)(NH_3)_{10}(SO_4)_2 + Co_4(N_2O_2)_2(NH_3)_{19}H_2O(SO_4)_4 + 28H_2O$$

These crystals are dissolved in 20% sulfuric acid and a 20% solution of nitric acid added. Bright rose red crystals separate. They are a double salt of pentamminonitrosocobalt nitrate with one sulfuric acid.

$$Co_2(N_2O_2)(NH_3)_{10}(SO_4)_2 + 4HNO_3 \rightarrow Co_2(N_2O_2)(NH_3)_{10}(NO_3)_4 \cdot H_2SO_4 + H_2SO_4$$

J. Sand and O. Genssler, Ann., **329**, 205 (1903)

20

$CoSO_4$

$NaBO_3$

KI

III-282

To volumetrically determine cobalt, the cobaltous salt is oxidized to cobaltic hydroxide (a), reduced by $KI + H_2SO_4$ (b) and the free iodine titrated against standardized thiosulfate (c).

(a) $$8CoSO_4 + 4NaBO_3 + 14NaOH + 5H_2O \rightarrow \underline{8Co(OH)_3} + Na_2B_4O_7 + 8Na_2SO_4$$

(b) $$2Co(OH)_3 + 2KI + 3H_2SO_4 \rightarrow 2CoSO_4 + I_2 + 6H_2O + K_2SO_4$$

(c) $$I_2 + 2Na_2S_2O_3 \rightarrow 2NaI + Na_2S_4O_6$$

H. H. Willard and D. Hall, J. Am. Chem. Soc., **44**, 2238 (1922)

1
16

$CoSO_4$

$NaC_{18}H_{33}O_2$ **III-283**

When an excess of cobalt sulfate is added to a solution of sodium oleate, a dark red cobalt oleate is formed. When nickel sulfate is used the precipitate consists of green resinous nickel oleate. When copper sulfate is used in the same way a precipitate of copper oleate is formed.

(a) $CoSO_4 + 2NaC_{18}H_{33}O_2 \rightarrow \underline{Co(C_{18}H_{33}O_2)_2} + Na_2SO_4$

(b) $NiSO_4 + 2NaC_{18}H_{33}O_2 \rightarrow \underline{Ni(C_{18}H_{33}O_2)_2} + Na_2SO_4$

(c) $CuSO_4 + 2NaC_{18}H_{33}O_2 \rightarrow \underline{Cu(C_{18}H_{33}O_2)_2} + Na_2SO_4$

L. Schön, Ann., **244**, 266 (1888)
Ref., L. Kahlenberg, J. Phys. Chem., **6**, 1 (1902) *7*

$CoSO_4$

$NaNH_4HPO_4$ **III-284**

Cobalt ammonium phosphate is precipitated by addition of sodium ammonium phosphate to cobaltous sulfate solution. The precipitate is converted to pyrophosphate by ignition.

(a) $CoSO_4 + NaNH_4HPO_4 \rightarrow \underline{CoNH_4PO_4} + (NaHSO_4)$

(b) $2CoNH_4PO_4 \rightarrow Co_2P_2O_7 + (2NH_3 + H_2O)$

T. Suzuki, J. Chem. Soc. (London), **97**, 727 (1910) *57*

$CoSO_4$

NaOCl **III-285**

Cobalt sulfate in aqueous solution precipitates cobaltic oxide when sodium hypochlorite is added.

$$2CoSO_4 + NaOCl + 2H_2O \rightarrow Co_2O_3 + NaCl + 2H_2SO_4$$

Rhodes and Hosking, Anal. Ed., **2**, 164 (1930) *44*
114
33

$CoSO_4$

NaOH **III-286**

Without access to air, cobaltous sulfate reacts with sodium hydroxide.

$$CoSO_4 + 2NaOH \rightarrow Co(OH)_2 + Na_2SO_4$$

Fremy, J. pharm. [3], **19**, 404 *25*

$CoSO_4$

$Na_2Fe(CN)_5 \cdot NO$ **III-287**

Cobalt nitroprusside is precipitated as a delicate pink colored powder when cobalt sulfate solution is added to a concentrated solution of sodium nitroprusside.

$$CoSO_4 + Na_2Fe(CN)_5 \cdot NO \rightarrow \underline{CoFe(CN)_5NO}(+Na_2SO_4)$$

T. H. Norton, Am. Chem. J., **10**, 223 (1888) *1*

$CoSO_4$

Na_2WO_4 **III-288**

Solutions of cobaltous sulfate and sodium tungstate are poured together and the precipitate washed and filtered.

$$CoSO_4 + Na_2WO_4 + 2H_2O \rightarrow \underline{CoWO_4 \cdot 2H_2O} + Na_2SO_4$$

Anthon, J. f. prakt. Chem., **9**, 344 (1836) *25*

$CoSO_4$

$Pb(CH_3 \cdot SO_4)_2$ **III-289**

Solutions of cobalt sulfate and lead methyl sulfate are mixed, the lead sulfate filtered off, and the filtrate yields leaf-like crystals of hexahydrated cobalt methyl sulfate.

$$CoSO_4 + Pb(CH_3SO_4)_2 + 6H_2O$$
$$\rightarrow \underline{PbSO_4} + Co(CH_3SO_4)_2 \cdot 6H_2O$$

J. E. Alèn, K. Sven. Vet. Ak. Handl. Övers., **37**, No. 8, 16 (1880) *10*

$CoSO_4$

PbO_2 **III-290**

Lead peroxide is added to a warm neutral solution of cobaltous sulfate.

$$4CoSO_4 + 4PbO_2 + 6H_2O \rightarrow \underline{4Co(OH)_3} + (4PbSO_4 + O_2)$$

Gibbs, Am. J. Sci., **14**, 204 (1852) *25*

$CoSO_4$

PbO_2 **III-291**

$K_2C_2O_4$

$HC_2H_3O_2$

A method for the determination of cobalt as trioxalatocobaltiate is based on the measurement of the absorption at 605 mμ due to the trioxalatocobaltiate ion, $Co(C_2O_4)_3^{---}$, which is produced by oxidation of a cobaltous solution by lead dioxide in a weakly acid solution of potassium oxalate according to the reaction.

$$2CoSO_4 + 4HC_2H_3O_2 + 7K_2C_2O_4 + PbO_2 \rightarrow 2K_3Co(C_2O_4)_3 + PbC_2O_4 + 4KC_2H_3O_2 + 2K_2SO_4 + 2H_2O$$

G. H. Cartledge and Parks M. Nichols, Anal. Ed., **13**, 20 (1941) *44*
16

$CoSO_4$

PbO_2 **III-292**

MnO_2

Cobalt oxide can be separated from the sulphate solution by boiling with either lead or manganese dioxides, or a mixture of the two.

(a) $2CoSO_4 + PbO_2 + H_2O \rightarrow Co_2O_3 + H_2SO_4 + PbSO_4$

(b) $3CoSO_4 + PbO_2 + 2H_2O \rightarrow Co_3O_4 + 2H_2SO_4 + PbSO_4$

(c) $2CoSO_4 + MnO_2 + H_2O \rightarrow Co_2O_3 + MnSO_4 + H_2SO_4$

(d) $2CoSO_4 + Mn_2O_3 \rightarrow Co_2O_3 + 2MnSO_4$

S. Field, Trans. Faraday Soc., **17**, 418 (1921) *85*

$CoSO_4$

Rb_2SO_4 **III-293**

Solutions containing molar quantities of cobalt sulfate and rubidium sulfate were mixed, stirred and allowed to stand and crystallize. An addition compound of cobalt rubidium disulfate was obtained. Similar addition products are formed when iron^{++}, cobalt, nickel or copper react with rubidium sulfate. In fact, similar addition products are formed when solutions of any of the above mentioned sulfates react with solutions of the sulfates of cesium, tellurium, ammonium or potassium.

$$CoSO_4 + Rb_2SO_4 + 6H_2O \rightarrow CoRb_2(SO_4)_2 \cdot 6H_2O$$

K. S. Krishman and A. Mookherji, Tran. Roy. Soc. (London), **237A**, 135 (1939) *105*

$CoSO_4$

$Sr(HC_4H_4O_5)_2$ **III-294**

Strontium hydrogen malate reacts with cobalt sulfate to form cobalt hydrogen malate. Treatment of this compound with alcohol converts it to the normal salt.

(a) $Sr(HC_4H_4O_5)_2 + CoSO_4 \rightarrow Co(HC_4H_4O_5)_2 + (SrSO_4)$

(b) $Co(HC_4H_4O_5)_2 \rightarrow CoC_4H_4O_5 + (H_2C_4H_4O_5)$

S. U. Pickering, J. Chem. Soc. (London), **107**, 946 (1915) *57*

$CoSO_4$

SrS_2O_3 **III-295**

A solution of cobaltous sulfate is treated with strontium thiosulfate, filtered, and the filtrate evaporated at a low temperature.

$$CoSO_4 + SrS_2O_3 + 6H_2O \rightarrow CoS_2O_3 \cdot 6H_2O + SrSO_4$$

Rammelsberg, Ann., **56**, 306 (1842) *25*

$CoSO_4$

Δ **III-296**

When heated at 800°C cobalt sulfate is decomposed:

$$CoSO_4 \rightarrow CoO + SO_3$$

G. Marchal, J. Chim. phys., **22**, 413 (1925) *69*

$Co(SCN)_2$

KNH_2 **III-297**

Slow addition of a liquid ammonia solution of potassium amide to a liquid ammonia solution of cobaltous thiocyanate at first precipitates bright blue flocculent cobaltous amide (a). On standing a few days in contact with liquid ammonia at ordinary temperatures, black amorphous cobaltous nitride is produced (b).

(a) $Co(SCN)_2 + 2KNH_2 \rightarrow \underline{Co(NH_2)_2} + 2KSCN$

(b) $3Co(NH_2)_2 \rightarrow Co_3N_2 + 4NH_3$

F. W. Bergstrom, J. Am. Chem. Soc., **46**, 2632 (1924) *1*

16

$CoSeO_3$

H_2SeO_3 **III-298**

Cobalt selenite dissolves in selenious acid, and the solution yields small, red, rhombic prisms of the anhydrous cobalt selenite selenium dioxide.

$$CoSeO_3 + H_2SeO_3 \rightarrow CoSeO_3 \cdot SeO_2 + H_2O$$

L. F. Nilson, K. Sven. Vet. Ak. Handl. Övers., **31**, No. 1, 41 (1874) *10*

$CoSeO_3$

SeO_2 **III-299**

H_2O

When normal cobalt selenite is heated in water with selenium dioxide at 60° mono-hydrated cobalt selenite diselenium dioxide forms red-violet, microscopic, oblique four-sided tablets.

$$CoSeO_3 + 2SeO_2 + H_2O \rightarrow CoSeO_3 \cdot (SeO_2)_2 \cdot H_2O$$

L. F. Nilson, Nova Acta. Soc. Uppsal. [3], **9**, No. 7, 57 (1874) *10*

$2CoCO_3 \cdot Co(OH)_2$

Δ **III-300**

Cobalt oxide is formed when basic cobalt carbonate is heated to 200°C.

$$2CoCO_3 \cdot Co(OH)_2 \rightarrow Co_3O_4 + 2CO_2 + H_2$$

C. D. Braun, Z. anal. Chem., **6**, 76 (1867) *28*

$Co_2(CO_3)_3$

KOH **III-301**

N_2O_3

Potassium cobaltinitrite is prepared by suspending cobalt carbonate in a small amount of water, adding the calculated amount of potassium hydroxide, and then passing in nitrogen trioxide.

$$Co_2(CO_3)_3 + 6KOH + 6N_2O_3 \rightarrow 2K_3Co(NO_2)_6 + 3CO_2 + 3H_2O$$

Rosenheim and Koppel, Z. anorg. Chem., **17**, 42 (1898)
Ref., M. Cunningham and F. M. Perkin, J. Chem. Soc. (London), **95:2**, 1563 (1909) *57*

$(CoCrO_4)_2 \cdot Cr_2O_7 \cdot 8NH_3$

$AgNO_3$ **III-302**

When a solution of chromato cobaltic dichromate octammoniate is treated with a solution of silver nitrate a precipitate of silver dichromate is formed and hydrated chromato cobaltic nitrate octammoniate will remain in solution.

$$(CoCrO_4)_2 \cdot Cr_2O_7 \cdot 8NH_3 + 2AgNO_3 + 2H_2O \rightarrow 2[CoNO_3 \cdot CrO_4 \cdot H_2O] \cdot 8NH_3 + \underline{Ag_2Cr_2O_7}$$

Samuel H. C. Briggs, J. Chem. Soc. (London), **115**:1, 74 (1919) *48*

$(CoCrO_4)_2 \cdot Cr_2O_7 \cdot 8NH_3$

$Ba(NO_3)_2$ **III-303**

When a solution of chromatocobalticdichromate octammoniate is treated with a solution of barium nitrate, a precipitate of barium chromate will be obtained and chromatocobaltic nitrateoctammoniate will remain in solution.

$$((CoCrO_4)_2Cr_2O_7 \cdot 8NH_3 + 2Ba(NO_3)_2 + 2H_2O \rightarrow \underline{2BaCrO_4} + (Co(NO_3)_2 \cdot CrO_4)_2 \cdot H_2O \cdot 8NH_3 + H_2))$$

Samuel H. C. Briggs, J. Chem. Soc. (London), **115**:1, 74 (1919) *48*

$Co_2Fe(CN)_6$

$K_4Fe(CN)_6$ **III-304**

Cobalt ferrocyanide boiled with potassium ferrocyanide gives the following reaction:

$$Co_2Fe(CN)_6 + 4K_4Fe(CN)_6 \rightarrow K_4Co_2(Fe(CN)_6) + 4K_3Fe(CN)_6$$

G. Wyrouboff, Gazz. chim. ital., **7**, 272 (1877) *21*

$Co_2H_6(C_2O_4)_6$

$CaCO_3$ **III-305**

The calcium salt of cobaltioxalic acid is obtained when a solution of the acid is treated with an excess of pure calcium carbonate and the solution agitated until a neutral or slightly acid reaction is obtained.

$$H_6Co_2(C_2O_4)_6 + 3CaCO_3 + 3H_2O \rightarrow Ca_3Co_2(C_2O_4)_6 \cdot 6H_2O + 3CO_2$$

Stanley B. Benedict, J. Am. Chem. Soc., **28**, 172 (1906) *1*

$Co(NH_3)_6(NO_2)_6Co$

HCl **III-306**

A solution of luteo cobaltocobaltic nitrite will react with diluted hydrochloric acid yielding a yellow precipitate.

$$Co(NH_3)_6(NO_2)_6Co + 6HCl \rightarrow \underline{CoCl_3(NH_3)_6} + 6HNO_2 + CoCl_3$$

S. M. Jörgensen, Z. anorg. Chem., **5**, 147 (1894) *28*

$Co(NH_3)_6(NO_2)_6Co$

Tl_2SO_4 **III-307**

The dry powder of luteo cobaltocobaltic nitrite will react with a solution of thallium sulfate yielding a precipitate of red color.

$$2[Co(NH_3)_6(NO_2)_6Co] + 3Tl_2SO_4 \rightarrow \underline{2Co(NO_2)_6Tl_3} + [Co(NH_3)_6]_2(SO_4)_3$$

S. M. Jörgensen, Z. anorg. Chem., **5**, 147 (1894) *28*

$Co_2(NH_3)_{10}Cl_6$

$NaNO_2$ **III-308**

$NaNO_3$

Chloropentammino cobaltic chloride reacts with a mixture of sodium nitrite and nitrate to form flavocobaltic chloride and sodium chloride.

$$Co_2(NH_3)_{10}Cl_6 + 2NaNO_2 + 2NaNO_3 \rightarrow Co_2(NH_3)_{10}(NO_2)_2(NO_3)_2Cl_2 + 4NaCl$$

W. Gibbs, Ber., **6**, 831 (1873) *11*

$Co_2(NH_3)_{10}(NO_2)_2(NO_3)_4$

$Co_2(NH_3)_{10}(NO_2)_2Cl_4$ **III-309**

When equal molecular quantities of xantho cobaltic nitrate and xantho cobaltic chloride are dissolved together in hot water a new salt is formed, flavocobaltic chloride.

$$Co_2(NH_3)_{10}(NO_2)_2(NO_3)_4 + Co_2(NH_3)_{10}(NO_2)_2Cl_4 \rightarrow 2Co_2(NH_3)_{10}(NO_2)_2(NO_3)_2Cl_2$$

W. Gibbs, Ber., **6**, 831 (1873) *11*

$Co_2(NH_3)_{10}(SO_4)_2I_2$

Br **III-310**

Bromine (or chlorine) replaces the iodine in roseo cobaltous ammonium sulfate iodide and yields dihydrated roseo cobaltous sulfate bromide (or chloride) as red crystals.

(a) $Co_2(NH_3)_{10}(SO_4)_2I_2 + Br_2 + 2H_2O \rightarrow Co_2(NH_3)_{10}(SO_4)_2Br_2(H_2O)_2 + (I_2)$

(b) $Co_2(NH_3)_{10}(SO_4)_2I_2 + Cl_2 + 2H_2O \rightarrow Co_2(NH_3)_{10}(SO_4)_2Cl_2 \cdot (H_2O)_2 + (I_2)$

J. M. Krok, Acta Univ. Lund., **78**, No. 5, 16–17 (1871) *10*

$Co_2(NH_3)_{10}(SO_4)_2I_2$

HNO_3 **III-311**

Nitric acid acts on roseo cobaltous ammonia sulfate iodide and yields brick red, amorphous dihydrated roseo cobaltous ammonium nitrate.

$Co_2(NH_3)_{10}(SO_4)_2I_2 + 6HNO_3 + 2H_2O \rightarrow 2H_2SO_4 + 2HI + Co_2(NH_3)_{10}(NO_3)_6 \cdot (H_2O)_2$

J. M. Krok, Acta Univ. Lund., **78**, No. 5, 19 (1871) *10*

$Co_2(NH_3)_{12}(SO_4)_2I_2$

Cl **III-312**

Chlorine replaces the iodine in luteo cobaltous ammonia sulfate iodide and yields yellowish red, prismatic crystals of luteo cobaltous ammonium sulfate chloride.

$Co_2(NH_3)_{12}(SO_4)_2I_2 + Cl_2 \rightarrow Co_2(NH_3)_{12}(SO_4)_2 \cdot Cl_2 + I_2$

J. M. Krok, Acta Univ. Lund., **78**, No. 5, 7 (1871) *10*

$Co_2(NH_3)_{12}(SO_4)_2I_2$

HCl **III-313**

Luteo cobaltous ammonia iodide sulfate dissolves in hydrochloric (or nitric acid) to form luteo cobaltous ammonium chloride (or nitrate).

(a) $Co_2(NH_3)_{12}(SO_4)_2I_2 + 6HCl \rightarrow Co_2(NH_3)_{12}Cl_6 + (2H_2SO_4 + 2HI)$

(b) $Co_2(NH_3)_{12}(SO_4)_2I_2 + 6HNO_3 \rightarrow Co_2(NH_3)_{12}(NO_3)_6 + (2H_2SO_4 + 2HI)$

J. M. Krok, Acta Univ. Lund., **78**, No. 5, 5 (1871) *10*

$Co_2(NO_2)_6(KNO_2)_6 \cdot 3H_2O$

Δ **III-314**

Potassium cobaltinitrite is decomposed on heating to yield cobalt oxide, nitric oxide, potassium nitrate, potassium nitrite, and water.

$$Co_2(NO_2)_6(KNO_2)_6 \cdot 3H_2O \rightarrow Co_2O_3 + 6NO + 3KNO_3 + 3KNO_2 + 3H_2O$$

P. C. Ray, J. Chem. Soc. (London), **89**:1, 555 (1906) *108*

$Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O$

$AgNO_3$ **III-315**

An aqueous solution of the red dimer of cobalt dinitrate mononitroso pentammoniate hemihydrate treated with silver nitrate solution gives as a precipitate an addition product of cobalt dinitrate mononitroso pentammoniate dimer containing one molecule of silver nitrate.

$$Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O + AgNO_3 \rightarrow \underline{Co_2(NO_3)_4(NO)_2(NH_3)_{10}AgNO_3} + (H_2O)$$

J. Sand and O. Genssler, Ann., **329**, 196 (1903)
Ref., A. Werner and P. Karrer, Helv. Chim., **1**, 64 (1918) *78*

$Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O$

HBr **III-316**

When the red dimer of cobalt dinitrate mononitroso pentammoniate hemihydrate is dissolved in concentrated hydrobromic acid and allowed to stand a short time, cobalt tribromide pentammoniate precipitates.

$$Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O + 6HBr \rightarrow \underline{2CoBr_3 \cdot 5NH_3} + (4HNO_3 + N_2O + 2H_2O)$$

A. Werner and P. Karrer, Helv. Chim., **1**, 76 (1918) *78*

$Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O$

HCl **III-317**

When the red dimer of cobalt dinitrate mononitroso pentammoniate hemihydrate is dissolved in concentrated hydrochloric acid and allowed to stand a short time, cobalt trichloride pentammoniate precipitates.

$$Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O + 6HCl \rightarrow \underline{2CoCl_3 \cdot 5NH_3} + (4HNO_3 + N_2O + 2H_2O)$$

A. Werner and P. Karrer, Helv. Chim., **1**, 76 (1918) *78*

$Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O$

$HClO_4$ **III-318**

From an aqueous solution of the red dimer of cobalt dinitrate mononitroso pentammoniate hemihydrate thirty percent perchloric acid precipitates a red-brown addition product of cobalt diperchlorate mononitroso pentammoniate hemihydrate containing one molecule of perchloric acid.

(a) $Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O + 4HClO_4 \rightarrow (Co_2(ClO_4)_4(NO)_2(NH_3)_{10} \cdot H_2O + 4HNO_3)$

(b) $Co_2(ClO_4)_4(NO)_2(NH_3)_{10} \cdot H_2O + HClO_4 \rightarrow \underline{Co_2(ClO_4)_4(NO)_2(NH_3)_{10}HClO_4 \cdot H_2O}$

A. Werner and P. Karrer, Helv. Chim., **1**, 65 (1918) *78*

$Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O$

HIO_3 **III-319**

A warm aqueous solution of iodic acid is added to an aqueous solution of the red dimer of cobalt dinitrate mononitroso pentammoniate hemihydrate to give a red precipitate of an addition product of cobalt di-iodate mononitroso pentammoniate dimer and iodic acid.

$$Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O + 8HIO_3 \rightarrow \underline{Co_2(IO_3)_4(NO)_2(NH_3)_{10} \cdot 4HIO_3} + (4HNO_3 + H_2O)$$

A. Werner and P. Karrer, Helv. Chim., **1**, 75 (1918) *78*

$Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O$

HNO_3 **III-320**

Treatment of the red dimer of cobalt dinitrate mononitrosopentammoniate hemihydrate with 20% nitric acid gives an addition product of cobalt dinitrate mononitrosopentammoniate dimer containing one molecule of dinitrogen pentoxide.

$$Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O + 2HNO_3 \rightarrow Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot N_2O_5 + (2H_2O)$$

J. Sand and O. Genssler, Ann., **329**, 196 (1903)
Ref., A. Werner and P. Karrer, Helv. Chim., **1**, 64 (1918) *78*

$Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O$

$H_2C_2O_4$ **III-321**

An aqueous solution of oxalic acid is poured over crystals of the red dimer of cobalt dinitrate mononitrosopentammoniate hemihydrate and precipitates a yellow-brown addition product of cobalt monooxalate mononitrosopentammoniate sesquihydrate dimer and oxalic acid.

$$Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O + 6H_2C_2O_4 + 2H_2O \rightarrow \underline{Co_2(C_2O_4)_2(NO)_2(NH_3)_{10} \cdot 4H_2C_2O_4 \cdot 3H_2O} + (4HNO_3)$$

A. Werner and P. Karrer, Helv. Chim., **1**, 74 (1918) *78*

$Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O$

KBr **III-322**

A cold concentrated aqueous solution of the red dimer of cobalt dinitrate mononitrosopentammoniate hemihydrate poured over crystals of potassium (or sodium) bromide gives a yellow-brown precipitate of cobalt dibromide mononitrosopentammoniate sesquihydrate dimer. Several recrystallizations from potassium bromide solution are necessary for a homogeneous product.

$$Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O + 4KBr + 2H_2O \rightarrow \underline{Co_2Br_4(NO)_2(NH_3)_{10} \cdot 3H_2O} + (4KNO_3)$$

A. Werner and P. Karrer, Helv. Chim., **1**, 71 (1918) *78*

$Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O$

KI **III-323**

HNO_3

An aqueous solution of the red dimer of cobalt dinitrate mononitroso pentammoniate hemihydrate when treated with potassium iodide and nitric acid gives a precipitate of cobalt dinitrate monoiodide pentammoniate dimer containing one molecule of dinitrogen oxide.

$$Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O + 2KI + 2HNO_3 \rightarrow \underline{Co_2(NO_3)_4I_2(NH_3)_{10} \cdot N_2O} + (2H_2O + 2KNO_3)$$

J. Sand and O. Genssler, Ann., **329**, 196 (1903)
Ref., A. Werner and P. Karrer, Helv. Chim., **1**, 66 (1918) *78*

$Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O$

K_2CrO_4 **III-324**

Aqueous solutions of potassium chromate and the red dimer of cobalt dinitrate mononitroso pentammoniate hemihydrate are mixed to give a yellow-brown precipitate of the dimer of cobalt monochromate mononitroso pentammoniate dihydrate.

$$Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O + 2K_2CrO_4 + 3H_2O \rightarrow \underline{Co_2(CrO_4)_2(NO)_2(NH_3)_{10} \cdot 4H_2O} + (4KNO_3)$$

A. Werner and P. Karrer, Helv. Chim., **1**, 75 (1918) *78*

$Co_2(NO_3)_4(NO)_2(NH_3)_{10}HNO_3 \cdot H_2O$

NH_4OH **III-325**

Crystals of the nitric acid addition product of cobalt dinitrate mononitrosopentammoniate hemihydrate dimer when triturated with concentrated ammonium hydroxide yield the neutral salt.

$$Co_2(NO_3)_4(NO)_2(NH_3)_{10}HNO_3 \cdot H_2O + NH_4OH \rightarrow Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O + (NH_4NO_3 + H_2O)$$

A. Werner and P. Karrer, Helv. Chim., **1**, 74 (1918) *78*

$Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O$

$Pb(NO_3)_2$ **III-326**

An aqueous solution of the red dimer of cobalt dinitrate mononitroso pentammoniate hemihydrate treated with a solution of lead nitrate gives as a precipitate an addition product of two molecules of cobalt dinitrate mononitrosopentammoniate dimer and one molecule of lead nitrate.

$$2Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O + Pb(NO_3)_2 \rightarrow \underline{Co_4(NO_3)_8(NO)_4(NH_3)_{20} \cdot Pb(NO_3)_2} + (2H_2O)$$

J. Sand and O. Genssler, Ann., **329**, 196 (1903)
Ref., A. Werner and P. Karrer, Helv. Chim., **1**, 57 (1918) *78*

$Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O$

Nitron Sulfate **III-327**

An aqueous solution of nitron sulfate is added to a solution of the red dimer of cobalt dinitrate mononitrosopentammoniate hemihydrate. After filtering off the precipitated nitron nitrate, addition of alcohol and ether precipitates the dimer of cobalt monosulfate mononitroso pentammoniate monohydrate.

$$2C_{20}H_{16}N_4 \cdot H_2SO_4 + Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O + H_2O \rightarrow Co_2(SO_4)_2(NO)_2(NH_3)_{10} \cdot 2H_2O + \underline{2C_{20}H_{16}N_4 \cdot HNO_3} + (2HNO_3)$$

A. Werner and P. Karrer, Helv. Chim., **1**, 69 (1918) *78*

$Co_2(N_2O_2)(NH_3)_{10}(NO_3)_4$

$HClO_4$ **III-328**

An aqueous solution of pentamminenitrosocobalt nitrate is treated with perchloric acid. The solution is concentrated and pale reddish yellow crystals separate. These are washed with dilute perchloric acid solution, alcohol and ether. The compound is pentamminenitrosocobalt chlorate, crystallizing with $2HNO_3$.

$$Co_2(N_2O_2)(NH_3)_{10}(NO_3)_4 + 4HClO_4 \rightarrow Co_2(N_2O_2)(NH_3)_{10}(ClO_4)_4 \cdot 2HNO_3 + 2HNO_3$$

J. Sand and O. Genssler, Ann., **329**, 202 (1903) *20*

$Co_2(N_2O_2)(NH_3)_{10}(NO_3)_4$

HNO_3 **III-329**

One gram of pentamminonitrosocobalt nitrate is dissolved in water and a 20% solution of nitric acid is added. Glistening plates separate. These are filtered off, washed with dilute nitric acid, alcohol and water. The compound is a double compound of pentaminonitrosocobalt nitrate and nitric acid.

$$Co_2(N_2O_2)(NH_3)_{10}(NO_3)_4 + 2HNO_3 \rightarrow Co_2(N_2O_2)(NH_3)_{10}(NO_3)_4 \cdot 2HNO_3$$

J. Sand and O. Genssler, Ann., **329**, 201 (1903) *20*

$(CoNi)_2S_4$

S_2Cl_2 **III-330**

Linnaeite is completely decomposed by sulfur monochloride when a mixture is heated in a sealed tube at 170°.

$$(CoNi)_2S_4 + 4S_2Cl_2 \rightarrow 2CoCl_2 + 2NiCl_2 + 6S_2$$

E. F. Smith, J. Am. Chem. Soc., **20**, 292 (1898) *1*

$Co_2(OH)_2Cl_4 \cdot 8NH_3$

HCl **III-331**

Dihydroxo dicobalt tetrachloride octammoniate reacts with concentrated hydrochloric acid at low temperature to yield dihydroxocobaltic chloride tetrammoniate and cobaltic chloride tetrammoniate.

$$(Co_2(OH)_2Cl_4 \cdot 8NH_3 + 2HCl \rightarrow Co(OH)_2Cl_2 \cdot 4NH_3 + CoCl_3 \cdot 4NH_3 + H_2)$$

A. Werner, Ber., **40**, 4817 (1907)
Ref., J. J. Sudborough, J. Chem. Soc. (London), **94:2**, 42 (1908) *57*

Co_2O_3

$FeSO_4$ **III-332**

Cobaltic oxide is treated with ferrous sulfate solution to produce ferric oxide and cobaltous sulfate which precipitates.

$$Co_2O_3 + 2FeSO_4 \rightarrow \underline{2CoSO_4} + Fe_2O_3$$

Goutal, Ann. chim. anal., **1**, 305 (1896) *76*

Co_2O_3

HCl **III-333**

Chlorine is liberated when cobalt sesquioxide is treated with hydrochloric acid.

$$Co_2O_3 + 6HCl \rightarrow 2CoCl_2 + 3H_2O + Cl_2$$

Donath, Ber. (1879) p. 1868
Ref., J. Am. Chem. Soc., **20**, 184 (1898) *1*

Co_2O_3

H_2 **III-334**

Cobaltic oxide is reduced by hydrogen at 2500°C.

$$Co_2O_3 + 3H_2 \rightarrow 2Co + 3H_2O$$

Moissan, Ann. chim. phys. [5], **21**, 199 (1880) *1*
25

Co_2O_3

H_2S **III-335**

When a current of hydrogen sulfide is passed over cobaltic oxide heated to redness, cobaltic sulfide separates in the form of globules, brass-yellow in color, with a metallic luster.

$$Co_2O_3 + 3H_2S \rightarrow Co_2S_3 + 3H_2O$$

T. Hjortdahl, Compt. rend., **65**, 76 (1867) *29*

Co_2O_3

K **III-336**

NH_3

At 0° cobaltic oxide is reduced first to cobaltous oxide (a) and then to elementary cobalt (b) by liquid ammonia solutions of potassium.

(a) $$Co_2O_3 + 2K + NH_3 \rightarrow 2CoO + KOH + KNH_2$$

(b) $$CoO + 2K + NH_3 \rightarrow Co + KOH + KNH_2$$

T. E. Moore and G. W. Watt, J. Am. Chem. Soc., **64**, 2774 (1942) *16*

$Co_2P_2O_7$

H_2O **III-337**

Cobalt pyrophosphate is heated in a sealed tube for six hours at 280°C.

$$2Co_2P_2O_7 + 2H_2O \rightarrow Co(H_2PO_4)_2 + \underline{Co_3(PO_4)_2}$$

Reynoso, Compt. rend., **34**, 795 (1852)

26

$Co_2(SO_3)_3 \cdot 5NH_3$

HCl

HBr **III-338**

HNO_3

Cobaltic sulfite pentammoniate treated with hydrochloric acid yields cobaltic chlorosulfite pentammoniate, water and sulfur dioxide. The corresponding bromo and nitrato sulfite pentammoniates are made similarly.

(a) $Co_2(SO_3)_3 \cdot 5NH_3 + 2HCl \rightarrow 2CoClSO_3 \cdot 5NH_3 + (H_2O + SO_2)$

(b) $Co_2(SO_3)_3 \cdot 5NH_3 + 2HBr \rightarrow 2CoBrSO_3 \cdot 5NH_3 + (H_2O + SO_2)$

(c) $Co_2(SO_3)_3 \cdot 5NH_3 + 2HNO_3$
$\rightarrow 2CoNO_3SO_3 \cdot 5NH_3 + (H_2O + SO_2)$

E. H. Riesenfeld, Med. K. Vet. Nobelinst., **6**, No. 6, 3 (1923)

10

$Co_2(SO_4)_2(NO)_2(NH_3)_{10} \cdot 2H_2O$

HBr **III-339**

A paste of cobalt monosulfate mononitroso pentammoniate monohydrate dimer and water is treated with hydrobromic acid (sp. gr. = 1.78). The product is an addition product of cobalt dibromide mononitroso pentammoniate hemihydrate dimer containing one molecule of hydrobromic acid.

$$Co_2(SO_4)_2(NO)_2(NH_3)_{10} \cdot 2H_2O + 5HBr$$
$$\rightarrow Co_2Br_4(NO)_2(NH_3)_{10}HBr \cdot H_2O + (2H_2SO_4 + H_2O)$$

A. Werner and P. Karrer, Helv. Chim., **1**, 72 (1918)

78

$Co_2(SO_4)_2(NO)_2(NH_3)_{10} \cdot 2H_2O$

HCl **III-340**

When the dimer of cobalt monosulfate mononitrosopentammoniate monohydrate is dissolved in concentrated hydrochloric acid and allowed to stand a short time, cobalt trichloride pentammoniate precipitates.

$$Co_2(SO_4)_2(NO)_2(NH_3)_{10} \cdot 2H_2O + 6HCl \rightarrow \underline{2CoCl_3 \cdot 5NH_3} + (2H_2SO_4 + N_2O + 3H_2O)$$

A. Werner and P. Karrer, Helv. Chim., **1**, 76 (1918) *78*

$Co_2(SO_4)_2(NO)_2(NH_3)_{10} \cdot 2H_2O$

HNO_3 **III-341**

To a solution of the red dimer of cobalt monosulfate mononitroso pentammoniate monohydrate in ten percent sulfuric acid is added a twenty percent solution of nitric acid. An orange-red precipitate of an addition product of cobalt dinitrate mononitrosopentammoniate hemihydrate dimer containing one molecule of nitric acid is formed.

(a) $Co_2(SO_4)_2(NO)_2(NH_3)_{10} \cdot 2H_2O + 4HNO_3 \rightarrow (Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O + 2H_2SO_4 + H_2O)$

(b) $Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O + HNO_3 \rightarrow \underline{Co_2(NO_3)_4(NO)_2(NH_3)_{10}HNO_3 \cdot H_2O}$

A. Werner and P. Karrer, Helv. Chim., **1**, 70 (1918) *78*

$Co_2(SO_4)_3$

Cs_2SO_4 **III-342**

Cobalt cesium alum is formed when strong sulfuric acid solutions of cobalt and cesium sulfates are electrolyzed.

$$Co_2(SO_4)_3 + Cs_2SO_4 + 24H_2O \rightarrow 2CoCs(SO_4)_2 \cdot 12H_2O$$

J. L. Howe and E. A. O'Neal, J. Am. Chem. Soc., **20**, 762 (1898) *1*

$Co_2(SO_4)_3$

$(NH_4)_2SO_4$ **III-343**

Cobaltic sulfate in solution prepared from cobaltous sulfate by electrolysis is treated with the theoretical amount of ammonium sulfate while the current continues to pass.

$$Co_2(SO_4)_3 + (NH_4)_2SO_4 + 24H_2O \rightarrow \underline{(NH_4)_2SO_4 \cdot Co_2(SO_4)_3 \cdot 24H_2O}$$

H. Marshall, J. Chem. Soc. (London), **59**, 768 (1891) *25*

$Co_2(SO_4)_3$

Rb_2SO_4 **III-344**

Cobalt rubidium alum is obtained when strong sulfuric acid solutions of cobalt sulfate and rubidium sulfate are electrolyzed.

$$Rb_2SO_4 + Co_2(SO_4)_3 + 24H_2O \rightarrow 2RbCo(SO_4)_2 \cdot 12H_2O$$

J. L. Howe and E. A. O'Neal, J. Am. Chem. Soc., **20**, 762 (1898) *1*

Co_3O_4

C **III-345**

When cobalt oxide is intimately mixed with carbon and subjected to temperatures ranging from 900°–1100°C, the cobalt oxide is reduced.

(a) $$Co_3O_4 + 4C \rightarrow 3Co + 4CO$$

(b) $$Co_3O_4 + 4CO \rightarrow 3Co + 4CO_2$$

(c) $$2Co_3O_4 + 4C \rightarrow 6Co + 4CO_2$$

H. T. Kalmus, J. Ind. Eng. Chem., **6**, 108 (1914) *22*

Co_3O_4

HCl **III-346**

Crude cobalt oxide dissolves in hydrochloric acid when heated and agitated with steam.

$$Co_3O_4 + 8HCl \rightarrow 3CoCl_2 + 4H_2O + Cl_2$$

H. T. Kalmus, J. Ind. Eng. Chem., **6**, 107 (1914) *22*

$[Co(NH_3)_3(NO_2)_3]$

$C_2H_8N_2$ **III-347**

10 Grams of cobaltic triammonotrinitrite are added to 31 grams of ethylenediamine and the mixture heated over a free flame. The reaction is rapid and ammonia is evolved. The temperature of the solution is raised to boiling and held there for 3–4 minutes, filtered and the filtrate concentrated until crystallization begins. The solution is cooled and the crystals removed by filtration. The compound formed is trinitrito ammono ethylenediamino cobaltic nitrate.

$$[Co(NH_3)_3(NO_2)_3] + C_2H_8N_2 \rightarrow [(NO_2)_3CoNH_3(C_2H_8N_2)] + 2NH_3$$

A. Werner and F. Chaussy, Ann., **386**, 254 (1912) *20*

$[Co(NH_3)_3(C_2O_4)OH]$

HBr **III-348**

3 Grams of hydroxooxalatotriammino cobalt are treated with 3 cc of concentrated hydrobromic acid while cooling. The residue is recrystallized from water by the addition of hydrobromic acid and oxalatoaquotriamminocobaltic bromide is obtained as crystals with one H_2O.

$$[(C_2O_4)OHCo(NH_3)_3] + HBr \rightarrow [(C_2O_4)H_2OCo(NH_3)_3]Br$$

A. Werner, E. Bindschedler and C. Sackur, Ann., **405**, 232 (1914) *20*

$[CoNH_3(NO_2)_3en]$

HCl **III-349**

10 Grams of ammonoethylenediamino cobaltic nitrite are rubbed with 80 cc of concentrated hydrochloric acid and heated for 3 minutes on the water bath. Nitrous acid is evolved and a black precipitate forms. The solution is allowed to stand for 20 hours at room temperature then the precipitate removed by filtration, washed with alcohol. It is then dissolved in a minimum volume of water, filtered and 25 cc of concentrated hydrochloric acid added. After 2 hours an additional 50 cc of acid is added and the solution allowed to stand 2 days. Small greenish black needles of dichloroaquo ammono ethylenediamino cobaltic chloride form. These are washed with dilute hydrochloric acid, alcohol and dried.

$$[(NO_2)_3CoNH_3C_2H_8N_2] + 3HCl + H_2O \rightarrow [Cl_2H_2OCoNH_3C_2H_8N_2]Cl + 3HNO_2$$

A. Werner and F. Chaussy, Ann., **386**, 254 (1912) *20*

$[Co(NH_3)_3C_2O_4OH]$

$H_2C_2O_4$ **III-350**

2 Grams of cobaltic hydroxooxalatotriammine, together with 1.5 grams of crystalline oxalic acid, are added to 20 cc of water and the solution evaporated to dryness on the water bath. The dark red residue is triturated several times with water and the red solution decanted from the residue. The residue is then washed with warm water until the extract does not react with a solution of calcium chloride. The residue is then dissolved in ammonia water by warming and dioxalatotriammine cobaltic acid liberated by the dropwise addition of dilute hydrochloric acid until the solution is neutral. The precipitate is filtered off, washed with alcohol and dried over sulphuric acid. The acid is amorphous, has a bluish red color and is insoluble in cold water. It dissolves in ammonia solution and is precipitated by mineral acids. The acid is insoluble in alcohol, ether and benzol. Yield 1.6 grams.

$$[CoOH(NH_3)_3C_2O_4] + H_2C_2O_4 \rightarrow [Co(NH_3)_3(C_2O_4)_2]H + H_2O$$

A. Werner and W. Bowis, Ann., **406**, 273 (1914) *20*

$[Co(NH_3)_3(NO_2)_3]$

$H_2C_2O_4$ **III-351**

5 Grams of cobaltic triammino nitrite and 5 grams of oxalic acid are dissolved in 40 cc of water and the solution boiled under reflux until all solid matter dissolves and a red precipitate forms (about 1 hour). The precipitate is recrystallized from hot water acidified with acetic acid and brown red prisms of trans triammino nitrito oxalate are formed.

$$[Co(NH_3)_3(NO_2)_3] + H_2C_2O_4 \rightarrow [Co(NH_3)_3NO_2(C_2O_4)] + 2HNO_2$$

A. Werner, E. Bindschedler and C. Sackur, Ann., **405**, 236 (1914) *20*

$[Co(NH_3)_6(NO_3)_3]$

$K_3[Cr(C_2O_4)_3]$ **III-352**

A solution of 0.7 grams of blue chromium potassium oxalate in 50 cc of water is added to one of 0.9 grams of hexamminocobaltnitrate in 80 cc of water. A grey precipitate forms and, after standing for some time, small brown crystals of hexamminocobalttrioxalatochromate with $3H_2O$ are formed. They are insoluble in water.

$$[Co(NH_3)_6(NO_3)_3] + [Cr(C_2O_4)_3]K_3 \rightarrow [Co(NH_3)_6 \cdot Cr(C_2O_4)_3] + 3KNO_3$$

P. Pfeiffer, Ann., **346**, 51 (1906) *20*

$[Co(NH_3)_3(C_2O_4)_2]H$

KOH **III-353**

3 Grams of dioxalatotriamminocobaltic acid are added to 10 cc of water and a solution of 0.52 grams of potassium hydroxide added. An equal volume of alcohol is added to the deep red solution and potassium dioxalatotriamminocobaltate is precipitated. The precipitate is washed with dilute then absolute alcohol and dried over sulphuric acid. It may be obtained in somewhat of a crystalline form by dissolving in water, adding alcohol to permanent turbidity, warming and allowing the solution to cool slowly. The crystals have two H_2O.

$$[Co(NH_3)_3(C_2O_4)_2]H + KOH \rightarrow \underline{[Co(NH_3)_3(C_2O_4)_2]K} + H_2O$$

A. Werner and W. Bowis, Ann., **406**, 274 (1914) *20*

$[Co(NH_3)_3(C_2O_4)_2]H$

NH_4OH **III-354**

3 Grams of dioxalatotriamminocobaltic acid ear added to 10 cc of water, stirred until an even suspension is formed and ammonium hydroxide added dropwise until solution is complete. An equal volume of alcohol is added and ammonium dioxalatotriamminocobaltate precipitates. It is washed with dilute, then absolute alcohol and dried over sulphuric acid. The compound is bluish red and crystallizes with two H_2O.

$$[Co(NH_3)_3(C_2O_4)_2]H + NH_4OH \rightarrow \underline{[Co(NH_3)_3(C_2O_4)_2]NH_4} + H_2O$$

A. Werner and W. Bowis, Ann., **406**, 275 (1914) *20*

$[Co(NH_3)_2(NO_2)_4]NH_4$

NH_3 **III-355**

A solution of ammonium diammino cobalt nitrite will react with ammonia at boiling temperature. If the solution is then slightly oversaturated with acetic acid, a yellow precipitate is obtained.

$$2NH_4[(NO_2)_2Co(NH_3)_2(NO_2)_2] + 2NH_3$$
$$\rightarrow \underline{(NO_2)_2 \cdot Co(NH_3)_2(NO_2)_2(NH_3)_4Co(NO_2)_2} + 2NH_4NO_2$$

S. M. Jörgensen, Z. anorg. Chem., **5**, 147 (1894) *28*

$[CoCO_3en_2]Br$

AgCl **III-356**

A concentrated aqueous solution of carbonatodiethylenediamine cobaltic bromide is shaken with excess silver chloride for a long time, the solution filtered and concentrated to crystallization. Bright red needles of carbonatodiethylenediamine cobaltic chloride with 1 H_2O deposit.

$$[CO_3Co(C_2H_8N_2)_2]Br + AgCl \rightarrow [CO_3Co(C_2H_8N_2)_2]Cl + (AgBr)$$

A. Werner and J. Rapiport, Ann., **386**, 73 (1912) *20*

$[CoCO_3en_2]Br$

$AgNO_3$ **III-357**

To a hot solution of carbonatodiethylenediamine cobaltic bromide is added an equimolar amount of $AgNO_3$. The precipitated AgBr is removed by filtration. When the filtrate is reduced to half its volume, bluish-red crystals of the corresponding nitrate are deposited.

$$[CO_3Co(C_2H_8N_2)_2]Br + AgNO_3 \rightarrow [CO_3Co(C_2H_8N_2)_2]NO_3 + \underline{AgBr}$$

A. Werner, Ann., **386**, 75 (1912) *41*

$[CoClNCSen_2]Br$

$AgNO_3$ **III-358**

A concentrated aqueous solution of trans chloroisothiocyanatodiethylenediamine cobaltic bromide is treated with a solution of 3 moles of silver nitrate and the solution heated to boiling. The solution is filtered and, upon cooling, bright violet needles deposit. These are washed with alcohol and ether. These crystals are trans chloro silver thiocyanatodiethylenediamine cobaltic nitrate.

$$[ClNCSCo(C_2H_8N_2)_2]Br + 2AgNO_3 \rightarrow [ClAgSCNCo(C_2H_8N_2)_2](NO_3)_2 + AgBr$$

A. Werner and R. Schmidt, Ann., **386**, 139 (1912) *20*

$[CoCO_3en_2]Br$

Ag_2O **III-359**

$C_3H_4O_4$

1 Gram of carbonatodiethylenediamine cobaltic bromide is dissolved in a small volume of water and 0.55 gram of freshly prepared silver oxide added. The solution is stirred until reaction is complete, then it is filtered and 2 moles of malonic acid added for each mole of the bromide used. The solution is concentrated on the water bath and rose colored crystals of malonatodiethylenediamine cobaltic acid malonate deposit. They are washed and dried over calcium chloride.

(a) $Ag_2O + H_2O \rightarrow 2AgOH$

(b) $[CO_3Co(C_2H_8N_2)_2]Br + AgOH$
$\rightarrow [CO_3Co(C_2H_8N_2)_2]OH + AgBr$

(c) $[CO_3Co(C_2H_8N_2)_2]OH + 2C_3H_4O_4$
$\rightarrow [(C_3H_2O_4Co(C_2H_8N_2)_2]C_3H_3O_4 + 2H_2O + CO_2$

A. Werner and J. Rapiport, Ann., **386**, 79 (1912) *20*

$[CoCO_3en_2]Br$

Ag_2O **III-360**

$S(CH_2COOH)_2$

Carbonatodiethylenediamine cobaltic bromide was treated with moist silver oxide and, after filtering, the solution was treated with thiodiacetic acid. After evaporating and cooling, a double salt containing cobaltous thiodiacetate precipitates.

(a) $2[CO_3Co(NH_2C_2H_4NH_2)_2]Br + Ag_2O + H_2O$
$\rightarrow 2[CO_3Co(NH_2C_2H_4NH_2)_2]OH + \underline{2AgBr}$

(b) $[CO_3Co(NH_2C_2H_4NH_2)_2]OH + 2S(CH_2COOH)_2$
$\rightarrow S(CH_2COO)_2Co \cdot S(CH_2COOH)_2 \cdot C_2H_4(NH_2)_2 \cdot 2H_2O + ?$

T. S. Price and S. A. Brazier, J. Chem. Soc. (London), **107**, 1371 (1915) *57*

$[CoClBren_2]Br$

HBr **III-361**

Cis chlorobromodiethylenediamine cobaltic bromide is treated with concentrated hydrobromic acid and the solution warmed gently until all of the solid has dissolved. The solution is cooled and a green crystalline mass precipitates. It is pressed on a porous plate and washed with alcohol. The residue is dissolved in a small volume of water and precipitated by the addition of ammonium bromide. Trans dibromodiethylenediamine cobaltic bromide is formed.

$$[ClBrCo(C_2H_8N_2)_2]Br + HBr \rightarrow [Br_2Co(C_2H_8N_2)_2]Br + HCl$$

A. Werner, L. Gerb, S. Lorie and J. Rapiport, Ann., **386**, 121 (1912) *20*

$[CoCO_3en_2]Br$

HBr **III-362**

Finely powdered carbonatodiethylenediamine cobaltic bromide is added to a colorless, fuming solution of hydrobromic acid and the mixture heated on the water bath until no more carbon dioxide is evolved. The blue violet reaction product is removed by filtration, washed free from acid with alcohol and pressed on a porous plate. The residue is added to ice cold water and the cis dibromodiethylenediamine cobaltic bromide remains as a residue.

$$[CO_3Co(C_2H_8N_2)_2]Br + 2HBr \rightarrow [Br_2Co(C_2H_8N_2)_2]Br + H_2O + CO_2$$

A. Werner, L. Gerb, S. Lorie and J. Rapiport, Ann., **386**, 114 (1912) *20*

$[CoCO_3en_2]Br$

HBr **III-363**

5 Grams of carbonato diethylenediamine cobaltic bromide are added to 20 cc of concentrated hydrobromic acid and the solution heated on the water bath with stirring until solution is complete and a green color is assumed by the solution. Upon cooling, the acid bromide precipitates. This is converted to the neutral bromide by heating the dry salt to 110°C until a portion dissolved in water does not react acid to litmus. 5 grams of the carbonato salt yield 5.7 grams of trans dibromodiethylenediamine cobaltic bromide.

(a) $[CO_3Co(C_2H_8N_2)_2]Br + 3HBr \rightarrow [Br_2Co(C_2H_8N_2)_2]Br \cdot HBr + H_2O + CO_2$

(b) $[Br_2Co(C_2H_8N_2)_2]Br \cdot HBr \rightarrow [Br_2Co(C_2H_8N_2)_2]Br + HBr$

A. Werner, L. Gerb, S. Lorie and J. Rapiport, Ann., **386**, 111 (1912) *20*

$[CoClNCSen_2]Br$

$HClO_4$ **III-364**

A solution of trans chloroisothiocyanatodiethylenediamine cobaltic bromide is treated with perchloric acid and violet crystals of trans chloroisothiocyanatodiethylenediamine cobaltic perchlorate precipitate. These may be recrystallized from hot water.

$$[ClNCSCo(C_2H_8N_2)_2]Br + HClO_4 \rightarrow [ClNCSCo(C_2H_8N_2)_2]ClO_4 + HBr$$

A. Werner and R. Schmidt, Ann., **386**, 136 (1912) *20*

$[CoCO_3en_2]Br$

HNO_3 **III-365**

NaBr

KOH

10 Grams of carbonatodiethylenediamine cobaltic bromide are mixed with 18 cc of cold water and 5 cc of concentrated nitric acid added. Carbon dioxide is evolved and the solution becomes yellowish red in color. This solution is neutralized with potassium hydroxide solution (1–5) and 5 cc excess added. 23 grams of solid sodium bromide are added and, in a short time, crystals of cis hydroxoaquodiethylenediamine cobaltic bromide deposit.

(a) $$[CO_3Co(C_2H_8N_2)_2]Br + 3HNO_3 \rightarrow [(NO_3)_2Co(C_2H_8N_2)_2]NO_3 + H_2O + CO_2 + HBr$$

(b) $$[(NO_3)_2Co(C_2H_8N_2)_2]NO_3 + 2NaBr + KOH + H_2O \rightarrow [OH(H_2O)Co(C_2H_8N_2)_2]Br_2 + 2NaNO_3 + KNO_3$$

A. Werner and K. Lange, Ann., **386**, 90 (1912) *20*

$[CoBrOHen_2]Br$

H_2O **III-366**

Cis hydroxoaquodiethylenediamine cobaltic bromide is shaken with a little water and the solution warmed. Red crystals of cis hydroxoaquodiethylenediamine cobaltic bromide deposit.

$$[BrOHCo(C_2H_8N_2)_2]Br + H_2O \rightarrow [H_2OOHCo(C_2H_8N_2)_2]Br_2$$

A. Werner and R. Schmidt, Ann., **386**, 131 (1912) *20*

$[CoBrNCSen_2]Br$

H_2O_2 **III-367**

3.5 Grams of trans bromoisothiocyanatodiethylenediamine cobaltic bromide are added to 10 cc of water, 1.5 cc of acetic acid, together with 5 cc of 30% hydrogen peroxide, are added and the solution warmed to 50–55°C and held there for 30 minutes. Solution is cooled and allowed to stand at room temperature for 5 hours. An equal volume of water and 5 grams of solid sodium dithionate are added. The solution is stirred until all solid material has dissolved. Bluish rose red crystals of trans bromoamminodiethylenediamino cobaltic dithionate precipitate as prism-like crystals. These are recrystallized by dissolving 1 gram in 30 cc water slightly acidified with acetic acid by heating. As the solution cools, fine bluish rose crystals of the compound deposit. These are washed twice with cold water and dried over calcium chloride.

(a) $[BrNCSCo(C_2H_8N_2)_2]Br + 2O_2 + 2H_2O \rightarrow [BrNH_3Co(C_2H_8N_2)_2]Br \cdot HSO_4 + CO_2$

(b) $[BrNH_3Co(C_2H_8N_2)_2]Br \cdot HSO_4 + Na_2S_2O_6 \rightarrow [BrNH_3Co(C_2H_8N_2)_2]S_2O_6 + NaBr + NaHSO_4$

A. Werner and W. Boës, Ann., **386**, 182 (1912) *20*

$[CoBr_2en_2]Br$

KI **III-368**

Cis dibromodiethylenediamine cobaltic bromide is dissolved in water to form a dilute solution and solid potassium iodide is added until crystals begin to form. Cis dibromodiethylenediamine cobaltic iodide deposits.

$$[Br_2Co(C_2H_8N_2)_2]Br + KI \rightarrow [Br_2Co(C_2H_8N_2)_2]I + KBr$$

A. Werner, L. Gerb, S. Lorie and J. Rapiport, Ann., **386**, 115 (1912) *20*

$[CoBr_2en_2]Br$

KSCN **III-369**

An aqueous solution of cis dibromodiethylenediamine cobaltic bromide is treated with solid potassium thiocyanate and a difficulty soluble precipitate forms. It is dissolved in cold water and solid potassium thiocyanate is added. Gray violet crystals of cis dibromodiethylenediamine cobaltic thiocyanate form with one H_2O.

$$[Br_2Co(C_2H_8N_2)_2]Br + KSCN \rightarrow [Br_2Co(C_2H_8N_2)_2]SCN + KBr$$

A. Werner, L. Gerb, S. Lorie and J. Rapiport, Ann., **386**, 116 (1912) *20*

$[CoBr_2en_2]Br$

KSCN **III-370**

$HClO_4$

2 Grams of trans dibromodiethylenediamine cobaltic bromide are added, in small portions, to 5 cc of water slightly warmed. The solution is stirred until all solid matter is dissolved then a solution of 0.46 grams of potassium thiocyanate in 2 cc of water is added and the solution heated. The precipitate which forms is a mixture of trans and cis bromoisothiocyanatodiethylenediamine cobaltic bromides. To prevent the formation of aquoisothiocyanato salts one must work quickly and be careful to heat the solution only until it is a blue violet color. The solution is cooled quickly, filtered and allowed to stand several hours. The mixture of trans and cis salts is triturated with perchloric acid and allowed to stand several hours then an excess of alcohol added. The precipitate is removed by filtration and washed with alcohol and ether. This residue is then treated with a small volume of water which dissolves the cis salt and forms a deep red colored solution. The less soluble trans salt remains as a residue.

(a) $[Br_2Co(C_2H_8N_2)_2]Br + KSCN$
$\rightarrow [BrNCSCo(C_2H_8N_2)_2]Br + KBr$

(b) $[BrNCSCo(C_2H_8N_2)_2]Br + HClO_4$
$\rightarrow [BrNCSCo(C_2H_8N_2)_2]ClO_4 + HBr$

A. Werner and R. Schmidt, Ann., **386**, 149 (1912) *20*

$[CoClBren_2]Br$

NH_4NO_3 **III-371**

Cis chlorobromodiethylenediamine cobaltic bromide is dissolved in cold water, quickly filtered and ammonium nitrate added. Small dark violet crystals of cis chlorobromodiethylenediamine cobaltic nitrate deposit. They are washed with alcohol and ether.

$[ClBrCo(C_2H_8N_2)_2]Br + NH_4NO_3$
$\rightarrow [ClBrCo(C_2H_8N_2)_2]NO_3 + NH_4Br$

A. Werner, L. Gerb, S. Lorie and J. Rapiport, Ann., **386**, 120 (1912) *20*

$[CoBr_2en_2]Br$

NH_4OH **III-372**

Trans dibromodiethylenediamino cobaltic bromide is covered with water, cooled in an ice bath and ammonium hydroxide (1:1) added dropwise with constant stirring until the color changes from green to dark violet. Cis bromoamminodiethylenediamine cobaltic bromide is formed. It may be precipitated from solution by the addition of hydrobromic acid and is obtained as dark brownish red needles. The reaction must be carried out at low temperatures otherwise both bromine atoms are replaced by ammonia and the diammono compound is formed.

$$[Br_2Co(C_2H_8N_2)_2]Br + NH_4OH \rightarrow [BrNH_3Co(C_2H_8N_2)_2]Br_2 + H_2O$$

A. Werner and W. Boes, Ann., **386**, 178 (1912) *20*

$[CoBr_2en_2]Br$

$NaNO_3$ **III-373**

A cold saturated aqueous solution of cis dibromodiethylenediamine cobaltic bromide is treated with solid sodium nitrate. The precipitate which forms is crystallized from cold water by the addition of solid sodium nitrate and small gray violet crystals of cis dibromodiethylenediamine cobaltic nitrate are obtained.

$$[Br_2Co(C_2H_8N_2)_2]Br + NaNO_3 \rightarrow [Br_2Co(C_2H_8N_2)_2]NO_3 + NaBr$$

A. Werner, L. Gerb, S. Lorie and J. Rapiport, Ann., **386**, 116 (1912) *20*

$[CoBrNCSen_2]Br$

$Na_2S_2O_6$ **III-374**

Cis bromoisothiocyanatodiethylenediamine cobaltic bromide is dissolved in the smallest possible volume of warm water and filtered. Solid sodium dithionate is added and the solution stirred to dissolve it. The solution is cooled quickly and the difficultly soluble cis bromoisothiocyanatodiethylenediamine cobaltic dithionate precipitates as brownish red crystals. A similar reaction occurs with the trans-form yielding violet blue leafy crystals.

$$2[BrNCSCo(C_2H_8N_2)_2]Br + Na_2S_2O_6 \rightarrow \underline{[BrNCSCo(C_2H_8N_2)_2]_2 \cdot S_2O_6} + 2NaBr$$

A. Werner and R. Schmidt, Ann., **386**, 152 (1912) *20*

$[CoBr_2en_2]Br$

$Na_2S_2O_6$ **III-375**

Solid sodium dithionate is added to a cold saturated aqueous solution of cis dibromodiethylenediamine cobaltic bromide and a precipitate of cis dibromodiethylenediamine cobaltic dithionate forms. It is purified by crystallizing from water. The color of this salt is a lighter violet than other salts of this series.

$$2[Br_2Co(C_2H_8N_2)_2]Br + Na_2S_2O_6 \rightarrow [Br_2Co(C_2H_8N_2)_2]_2S_2O_6 + 2NaBr$$

A. Werner, L. Gerb, S. Lorie and J. Rapiport, Ann., **386**, 117 (1912) *20*

$[CoClBren_2]Br$

$Na_2S_2O_6$ **III-376**

Cis chlorobromodiethylenediamine cobaltic bromide is dissolved in cold water, filtered quickly and solid sodium dithionate added. Cis chlorobromodiethylenediamine cobaltic dithionate deposits as small violet crystals which are only slightly soluble in water.

$$2[ClBrCo(C_2H_8N_2)_2]Br + Na_2S_2O_6 \rightarrow [ClBrCo(C_2H_8N_2)_2]_2S_2O_6 + 2NaBr$$

A. Werner, L. Gerb, S. Lorie and J. Rapiport, Ann., **386**, 120 (1912) *20*

$[CoClNCSen_2]Br$

$Na_2S_2O_6$ **III-377**

A concentrated aqueous solution of trans chloroisothiocyanatodiethylenediamine cobaltic bromide is treated with solid sodium dithionate and the soluton stirred until all of the solid dissolves. Difficultly soluble blue violet crystals of trans chloroisothiocyanatodiethylenediamine cobaltic dithionate deposit.

$$2[ClNCSCo(C_2H_8N_2)_2]Br + Na_2S_2O_6 \rightarrow [ClNCSCo(C_2H_8N_2)_2]_2S_2O_6 + 2NaBr$$

A. Werner and R. Schmidt, Ann., **386**, 135 (1912) *20*

$[Co(NH_3)_2Cl_2en]Br$

$Na_2S_2O_6$ **III-378**

Cis dichloroethylenediamino diammono cobaltic bromide is dissolved in cold water and solid sodium dithionate added. Violet cis dichloroethylenediaminodiammino cobaltic dithionate precipitates. It is filtered off and dried on a porous plate. This precipitate is impure so it is mixed with ammonium chloride and triturated to convert the salt to the chloride. This is then made into a thick paste by the addition of water and the suspension filtered. Cis salt is soluble and is in the filtrate from this suspension. Solid sodium dithionate is added to the filtrate and the dithionate is deposited.

$$2[Cl_2Co(C_2H_8N_2)(NH_3)_2]Br + Na_2S_2O_6 \rightarrow [Cl_2Co(C_2H_8N_2)(NH_3)_2]_2S_2O_6 + 2NaBr$$

A. Werner and F. Chaussy, Ann., **386**, 263 (1912) *20*

$[CoCO_3en_2]Br$

$SO_2(CH_2COOH)_2$ **III-379**

AgCl

KI

Carbonatodiethylenediamine cobaltic bromide solution reacts with sulfonyl diacetic acid to precipitate a complex bromide. This bromide is converted to the chloride by treatment with silver chloride, and to the iodide by treatment with potassium iodide.

(a) $$[CO_3Co(NH_2C_2H_4NH_2)_2]Br + SO_2(CH_2COOH)_2 + H_2O \rightarrow \underline{[SO_2(CH_2COO)_2Co(NH_2C_2H_4NH_2)_2]Br \cdot 2H_2O} + (CO_2)$$

(b) $$[SO_2(CH_2COO)_2Co(NH_2C_2H_4NH_2)_2]Br \cdot 2H_2O + AgCl \rightarrow [SO_2(CH_2COO)_2Co(NH_2C_2H_4NH_2)_2]Cl \cdot H_2O + \underline{(AgBr} + H_2O)$$

(c) $$[SO_2(CH_2COO)_2Co(NH_2C_2H_4NH_2)_2]Br \cdot 2H_2O + KI \rightarrow [SO_2(CH_2COO)_2Co(NH_2C_2H_4NH_2)_2]I + (KBr + 2H_2O)$$

T. S. Price and S. A. Brazier, J. Chem. Soc. (London), **107**, 1373 (1915) *57*

$[CoBr_2en_2]Br$

Δ **III-380**

An aqueous solution of 5 grams of trans dibromodiethylenediamine cobaltic bromide is evaporated to dryness on the water bath and the color changes to dark red and finally to a dark violet color. This is taken up in a little water and allowed to crystallize. Dark leafy crystals of the cis form of the compound form.

$$\text{trans } [Br_2Co(C_2H_8N_2)_2]Br \rightarrow \text{cis } [Br_2Co(C_2H_8N_2)_2]Br$$

A. Werner, L. Gerb, S. Lorie and J. Rapiport, Ann., **386**, 112 (1912) *20*

$[CoBr_2en_2]Br$

H_2O **III-381**

A concentrated aqueous solution of trans dibromodiethylenediamino cobaltic bromide is stirred while being warmed to 40°C. This is continued until the solution becomes a violet color then it is cooled and saturated with sodium bromide. A mixture of a violet salt and a green salt is precipitated. These may be separated by fractional solution when the violet salt, cis bromoaquodiethylenediamino cobaltic bromide remains as a residue due to its slight solubility.

$$[Br_2Co(C_2H_8N_2)_2]Br + H_2O \rightarrow [BrH_2OCo(C_2H_8N_2)_2]Br_2$$

A. Werner and R. Schmidt, Ann., **386**, 127 (1912) *20*

$[CoSO_2(CH_2COO)_2en_2]Br \cdot 2H_2O$

$AgNO_3$ **III-382**

KCNS

Sulfonyldiacetodiethylenediamine cobaltic bromide is converted to the nitrate by treating with silver nitrate and to the thiocyanate by treatment with potassium thiocyanate.

(a) $[SO_2(CH_2COO)_2Co(NH_2C_2H_4NH_2)_2]Br \cdot 2H_2O + AgNO_3$
$\rightarrow [SO_2(CH_2COO)_2Co(NH_2C_2H_4NH_2)_2]NO_3 \cdot 2H_2O + \underline{(AgBr)}$

(b) $[SO_2(CH_2COO)_2Co(NH_2C_2H_4NH_2)_2]Br \cdot 2H_2O + KCNS$
$\rightarrow [SO_2(CH_2COO)_2Co(NH_2C_2H_4NH_2)_2]CNS + (KBr + 2H_2O)$

T. S. Price and S. A. Brazier, J. Chem. Soc. (London), **107**, 1375 (1915) *57*

$[CoNH_3NO_2en_2]Br_2$

AgCl **III-383**

An aqueous solution of cis nitritoamminodiethylenediamino cobaltic bromide saturated at 30°C is triturated with freshly prepared silver chloride. The solution is filtered and allowed to concentrate over sulfuric acid. Cis nitritoamminodiethylenediamino cobaltic chloride crystallizes as orange yellow prisms.

$$[NO_2NH_3Co(C_2H_8N_2)_2]Br_2 + 2AgCl \rightarrow [NO_2NH_3Co(C_2H_8N_2)_2]Cl_2 + 2AgBr$$

A. Werner and W. Boes, Ann., **386**, 219 (1912) *20*

$[CoNH_3NO_2en_2]Br_2$

$AgNO_3$ **III-384**

A one fourth saturated aqueous solution of cis nitritoamminodiethylenediamino cobaltic bromide is treated with an aqueous solution of silver nitrate until no more precipitate forms. Several drops of acetic acid is added, the solution filtered and allowed to concentrate at ordinary temperature. Yellowish brown crystals of cis nitritoamminodiethylenediamino cobaltic nitrate deposit.

$$[NO_2NH_3Co(C_2H_8N_2)_2]Br_2 + 2AgNO_3 \rightarrow [NO_2NH_3Co(C_2H_8N_2)_2](NO_3)_2 + 2AgBr$$

A. Werner and W. Boes, Ann., **386**, 220 (1912) *20*

$[CoH_2O(OH)en_2]Br_2$

CO_2

KOH **III-385**

5 Grams of hydroxoaquodiethylenediamine cobaltic bromide are added to 13.6 cc of normal potassium hydroxide and the mixture warmed until solution is complete. The solution is cooled and carbon dioxide passed into it for one hour. A red crystalline mass precipitates and solid potassium bromide is added to salt out the compound. The precipitate is recrystallized from water containing potassium bromide and carbonatodiethylenediamine cobaltic bromide is obtained as red needles.

$$[(H_2O)OHCo(C_2H_8N_2)_2]Br_2 + CO_2 + KOH \rightarrow [CO_3Co(C_2H_8N_2)_2]Br + 2H_2O + KBr$$

A. Werner and R. Bosshard, Ann., **386**, 101 (1912) *20*

$[CoH_2OClen_2]Br_2$

HBr **III-386**

2 Grams of cis chlorodiethylenediamino cobaltic bromide are added to 2 cc of concentrated hydrobromic acid and heated until solution is complete. The green solution is cooled quickly and allowed to stand. A mass of crystals soon deposits. This is a mixture of the green trans and cis chlorobromodiethylenediamine cobaltic bromides. The precipitate is filtered off, washed with alcohol and ether then dried. The dry mixture is extracted with small portions of water and the green trans salt dissolves. The cis salt may be recovered from the residual solution. This violet cis salt is washed with alcohol and dried.

$$[Cl(H_2O)Co(C_2H_8N_2)_2]Br_2 \rightarrow [ClBrCo(C_2H_8N_2)_2]Br + H_2O$$

A. Werner, L. Gerb, S. Lorie and J. Rapiport, Ann., **386**, 118 (1912) *20*

$[CoH_2O(OH)en_2]Br_2$

HBr **III-387**

16 grams of hydroxoaquodiethylenediamine cobaltic bromide are triturated with concentrated hydrobromic acid, the residue pressed on a porous plate and washed with alcohol and ether. The residue is dissolved in 35 cc of water with the aid of a few drops of hydrobromic acid, cooled to 0°C and saturated with hydrobromic acid. Cis diaquodiethylenediamine cobaltic bromide crystallizes as brown red crystals.

$$[(H_2O)OHCo(C_2H_8N_2)_2]Br_2 + HBr \rightarrow [(H_2O)_2Co(C_2H_8N_2)_2]Br_3$$

A. Werner and K. Lange, Ann., **386**, 90 (1912) *20*

$[CoH_2O(OH)en_2]Br_2$

HBr **III-388**

Cis hydroxoaquodiethylenediamine cobaltic bromide is triturated with concentrated hydrobromic acid and cis diaquodiethylenediamine cobaltic bromide is formed.

$$[(H_2O)OHCo(C_2H_8N_2)_2]Br_2 + HBr \rightarrow [(H_2O)_2Co(C_2H_8N_2)_2]Br_3$$

A. Werner and R. Schmidt, Ann., **386**, 131 (1912) *20*

$[CoNH_3Bren_2]Br_2$

$HClO_4$ **III-389**

1 Gram of trans bromoamminodiethylenediamino cobaltic bromide is dissolved in a small volume of water and a few drops of perchloric acid added. Crystals begin to form in a short time and the solution is allowed to stand for 1 hour. The residue is recrystallized from warm water and violet needles of trans bromoamminodiethylenediamino cobaltic perchlorate deposit.

$$[BrNH_3Co(C_2H_8N_2)_2]Br_2 + 2HClO_4 \rightarrow [BrNH_3Co(C_2H_8N_2)_2](ClO_4)_2 + 2HBr$$

A. Werner and W. Boes, Ann., **386**, 184 (1912) *20*

$[CoNH_3Bren_2]Br_2$

HNO_3 **III-390**

Finely pulverized cis bromoamminodiethylenediamino cobaltic bromide is suspended in water and concentrated nitric acid added, with stirring, until a thick crystalline mass forms. Two drops of water are added followed by 6–10 drops of nitric acid. This operation is repeated until half of the residue is dissolved. The solution is warmed on the water bath until solution is complete then filtered. The filtrate is cooled and the residue which precipitates is recrystallized from water. Dark red violet to black crystals of cis bromoamminodiethylenediamino cobaltic nitrate deposit.

$$[BrNH_3Co(C_2H_8N_2)_2]Br_2 + 2HNO_3 \rightarrow [BrNH_3Co(C_2H_8N_2)_2](NO_3)_2 + 2HBr$$

A. Werner and W. Boes, Ann., **386**, 180 (1912) *20*

$[CoNH_3NO_2en_2]Br_2$

HNO_3 **III-391**

Concentrated nitric acid is added dropwise to a cold saturated aqueous solution of cis nitritoamminodiethylenediamino cobaltic bromide and a crystalline precipitate forms. This is filtered off, dissolved in water and the solution allowed to concentrate slowly. Reddish brown lustrous prisms of cis nitritoamminodiethylenediamino cobaltic nitrate deposit.

$$[NO_2NH_3Co(C_2H_8N_2)_2]Br_2 + 2HNO_3 \rightarrow [NO_2NH_3Co(C_2H_8N_2)_2](NO_3)_2 + 2HBr$$

A. Werner and W. Boes, Ann., **386**, 220 (1912) *20*

$[CoNH_3NO_2en_2]Br_2$

H_2SO_4 **III-392**

A saturated aqueous solution of cis nitritoamminodiethylenediamino cobaltic bromide is treated with an equal volume of concentrated sulfuric acid. The solution is allowed to stand 1 hour then alcohol and ether added until a voluminous precipitate forms. This is removed by suction filtration, dissolved in water and the solution allowed to concentrate slowly. Long bright yellow prismatic needles of cis nitritoamminodiethylenediamino cobaltic sulphate are deposited.

$$[NO_2NH_3Co(C_2H_8N_2)_2]Br_2 + H_2SO_4 \rightarrow [NO_2NH_3Co(C_2H_8N_2)_2]SO_4 + 2HBr$$

A. Werner and W. Boes, Ann., **386**, 222 (1912) *20*

$[CoH_2O(OH)en_2]Br_2$

KI **III-393**

2 Grams of trans hydroxoaquodiethylenediamine cobaltic bromide are dissolved in 50 cc of water and 10 grams of solid potassium iodide added. Reddish brown monoclinic crystals of trans hydroxoaquodiethylenediamine cobaltic iodide deposit.

$$[(H_2O)OHCo(C_2H_8N_2)_2]Br_2 + 2KI \rightarrow [(H_2O)OHCo(C_2H_8N_2)_2]I_2 + 2KBr$$

A. Werner and K. Lange, Ann., **386**, 97 (1912) *20*

$[CoNH_3NO_2en_2]Br_2$

KI **III-394**

A solution of cis nitritoamminodiethylenediamino cobaltic bromide, saturated at room temperature, is treated with solid potassium iodide until no more dissolves. Cis nitritoamminodiethylenediamino cobaltic iodide precipitates almost quantitatively in small yellow needles. These are filtered off, sucked dry, dissolved in water at 25°C and alcohol added to recrystallize.

$$[NO_2NH_3Co(C_2H_8N_2)_2]Br_2 + 2KI \rightarrow [NO_2NH_3Co(C_2H_8N_2)_2]I_2 + 2KBr$$

A. Werner and W. Boes, Ann., **386**, 219 (1912) *20*

$[CoH_2ONCSen_2]Br_2$

KSCN **III-395**

A concentrated aqueous solution of trans aquoisothiocyanatodiethylenediamine cobaltic bromide is treated with solid potassium thiocyanate and a violet precipitate forms. It is recrystallized from a concentrated aqueous solution by the addition of potassium thiocyanate and trans aquoisothiocyanatodiethylenediamine cobaltic thiocyanate deposits. The crystals have one H_2O. Heating the compound at 100°C transforms it into diisothiocyanatodiethylenediamine cobaltic thiocyanate.

(a) $[H_2ONCSCo(C_2H_8N_2)_2]Br_2 + 2KSCN \rightarrow [H_2ONCSCo(C_2H_8N_2)_2](SCN)_2 + 2KBr$

(b) $[H_2ONCSCo(C_2H_8N_2)_2](SCN)_2 \rightarrow [(NCS)_2Co(C_2H_8N_2)_2](SCN) + H_2O$

A. Werner and R. Schmidt, Ann., **386**, 160 (1912)

20

$[CoH_2O(OH)en_2]Br_2$

KSCN **III-396**

An aqueous solution of trans hydroxoaquodiethylenediamine cobaltic bromide is treated with solid potassium thiocyanate and brown hydroxoaquodiethylenediamine cobaltic thiocyanate, the trans form, precipitates.

$[(H_2O)OHCo(C_2H_4N_2)_2]Br_2 + 2KSCN \rightarrow \underline{[(H_2O)OHCo(C_2H_8N_2)_2](SCN)_2} + 2KBr$

A. Werner and K. Lange, Ann., **386**, 98 (1912)

20

$[CoNH_3Bren_2]Br_2$

K_2PtCl_4 **III-397**

A concentrated aqueous solution of potassium chloroplatinite is added to a solution of cis bromoamminodiethylenediamino cobaltic bromide and reddish brown lustrous crystals of cis bromoamminodiethylenediamino cobaltic chloroplatinite precipitate.

$[BrNH_3Co(C_2H_8N_2)_2]Br_2 + K_2PtCl_4 \rightarrow [BrNH_3Co(C_2H_8N_2)_2]PtCl_4 + 2KBr$

A. Werner and W. Boes, Ann., **386**, 182 (1912)

20

$[CoH_2OClen_2]Br_2$

$LiNO_3$ **III-398**

3 Grams of cis chloroaquodiethylenediamine cobaltic bromide are dissolved in cold water and 7 grams of powdered lithium nitrate added. The mixture is stirred until solution is complete. After a short time a reddish brown precipitate forms which is removed by filtration, dissolved in 30 cc of water and 7 grams of lithium nitrate added. Cis chloroaquodiethylenediamine cobaltic bromonitrate precipitates.

$$[Cl(H_2O)Co(C_2H_8N_2)_2]Br_2 + LiNO_3 \rightarrow \underline{[Cl(H_2O)Co(C_2H_8N_2)_2]BrNO_3} + LiBr$$

A. Werner, L. Gerb, S. Lorie and J. Rapiport, Ann., **386**, 124 (1912) *20*

$[CoNH_3Bren_2]Br_2$

$LiNO_3$ **III-399**

Trans bromoamminodiethylenediamino cobaltic bromide is dissolved in a small volume of water, filtered, and solid lithium nitrate added to the filtrate. The solution is allowed to stand a short time and crystals of trans bromoamminodiethylenediamino cobaltic nitrate with one H_2O form. These are washed with alcohol and ether then dried over calcium chloride. The salt dissolves easily in water.

$$[BrNH_3Co(C_2H_8N_2)_2]Br_2 + 2LiNO_3 \rightarrow [BrNH_3Co(C_2H_8N_2)_2](NO_3)_2 + 2LiBr$$

A. Werner and W. Boes, Ann., **386**, 184 (1912) *20*

$[CoH_2OBren_2]Br_2$

NH_4OH **III-400**

5 Grams of bromoaquodiethylenediamine cobaltic bromide is added to 8 cc of a cold 22% solution of ammonium hydroxide. The blue bromoaquo bromide dissolves and, in a short time, a brownish violet precipitate forms which is quickly removed by filtration and washed with alcohol and ether. The yield of cis hydroxobromodiethylenediamine cobaltic bromide is 2.7 grams.

$$[BrH_2OCo(C_2H_8N_2)_2]Br_2 + NH_4OH \rightarrow [BrOHCo(C_2H_8N_2)_2]Br + NH_4Br + H_2O$$

A. Werner and R. Schmidt, Ann., **386**, 131 (1912) *20*

$[CoH_2OBren_2]Br_2$

$NaNO_2$ **III-401**

6 Grams of cis bromoaquodiethylenediamine cobaltic bromide are dissolved in 40 cc of cold water, the solution filtered and 17 grams of solid sodium nitrite added to the filtrate. Cis bromoaquodiethylenediamine cobaltic nitrite deposits as a blue violet crystalline powder. This is removed by filtration, dissolved in 16 cc of cold water and 8 grams of solid sodium nitrite added. The crystals which are deposited are washed with alcohol and water then dried over calcium chloride.

$$[Br(H_2O)Co(C_2H_8N_2)_2]Br_2 + 2NaNO_2 \rightarrow [Br(H_2O)Co(C_2H_8N_2)_2](NO_2)_2 + 2NaBr$$

A. Werner and R. Schmidt, Ann., **386**, 129 (1912) *20*

$[CoH_2OBren_2]Br_2$

$NaNO_3$ **III-402**

Cis aquo bromodiethylenediamine cobaltic bromide is dissolved in the minimum volume of water, filtered and solid sodium nitrate added until it is in excess. The mass is triturated and allowed to stand one hour. Violet cis bromoaquodiethylenediamine cobaltic nitrate forms. It is sucked dry and crystallized from water by the addition of sodium nitrate. Blue violet crystals with one H_2O are obtained.

$$[Br(H_2O)Co(C_2H_8N_2)_2]Br_2 + 2NaNO_3 \rightarrow [Br(H_2O)Co(C_2H_8N_2)_2](NO_3)_2 + 2NaBr$$

A. Werner and R. Schmidt, Ann., **386**, 129 (1912) *20*

$[CoNH_3Bren_2]Br_2$

$Na_2S_2O_6$ **III-403**

A saturated aqueous solution of cis bromoamminodiethylenediamino cobaltic bromide is treated with considerable excess of a concentrated aqueous solution of sodium dithionate and cis bromoamminodiethylenediamino cobaltic dithionate precipitates as a difficultly soluble red violet fine crystalline powder. It is washed with alcohol and ether.

$$[BrNH_3Co(C_2H_8N_2)_2]Br_2 + Na_2S_2O_6 \rightarrow [BrNH_3Co(C_2H_8N_2)_2]S_2O_6 + 2NaBr$$

A. Werner and W. Boes, Ann., **386**, 181 (1912) *20*

$[CoNH_3NO_2en_2]Br_2$

$Na_2S_2O_6$ **III-404**

A half saturated aqueous solution of sodium dithionate is added to a saturated aqueous solution of cis nitritoammonodiethylenediamino cobaltic bromide and cis nitritoammonodiethylenediamino cobaltic dithionate precipitates as a yellow powder.

$$[NO_2NH_3Co(C_2H_8N_2)_2]Br_2 + Na_2S_2O_6 \rightarrow [NO_2NH_3Co(C_2H_8N_2)_2]S_2O_6 + 2NaBr$$

A. Werner and W. Boes, Ann., **386**, 220 (1912) *20*

$[CoH_2O(OH)en_2]Br_2$

$Na_2S_2O_6$ **III-405**

5 Grams of solid sodium dithionate are added to a solution of 2 grams of trans hydroxoaquodiethylenediamine cobaltic bromide. Bright red trans hydroxoaquodiethylenediamine cobaltic dithionate precipitates. The salt is very soluble in water but insoluble in alcohol and ether.

$$[(H_2O)OHCo(C_2H_8N_2)_2]Br_2 + Na_2S_2O_6 \rightarrow \underline{[(H_2O)OHCo(C_2H_8N_2)_2](S_2O_6)} + 2NaBr$$

A. Werner and K. Lange, Ann., **386**, 98 (1912) *20*

$[CoH_2OClen_2]Br_2$

Δ **III-406**

Chloroaquodiethylenediamine cobaltic bromide is heated for two hours in an air oven at 110°C. A mixture of the cis and trans chlorobromodiethylenediamine cobaltic bromides is formed. These may be separated by extracting the mixture with very small volumes of water. The dark green trans salt is soluble but the violet cis form is not so soluble. The green trans chlorobromodiethylenediamine cobaltic bromide may be precipitated from its aqueous solution as the nitrate by the addition of ammonium nitrate.

$$[Cl(H_2O)Co(C_2H_8N_2)_2]Br_2 \rightarrow [ClBrCo(C_2H_8N_2)_2]Br + H_2O$$

A. Werner, L. Gerb, S. Lorie and J. Rapiport, Ann., **386**, 118 (1912) *20*

$[CoH_2OClen_2]Br_2$

Δ **III-407**

Chloroaquodiethylenediamine cobaltic bromide is heated to constant weight at 110°C, and the residue treated with concentrated hydrobromic acid. The reaction product is extracted with small portions of water until the green trans salt is removed. The residue is a gray violet crystalline compound which is dissolved in water and solid ammonium bromide added until precipitation begins. Cis chlorobromodiethylenediamine cobaltic bromide is formed.

$$[Cl(H_2O)Co(C_2H_8N_2)_2]Br_2 \rightarrow [ClBrCo(C_2H_8N_2)_2]Br + H_2O$$

A. Werner, L. Gerb, S. Lorie and J. Rapiport, Ann., **386**, 120 (1912) *20*

$[CoH_2OClen_2]Br_2$

Δ **III-408**

NH_4SCN

Chloroaquodiethylenediamine cobaltic bromide is heated to constant weight at 110°C, extracted with cold water and then treated with a solution of ammonium thiocyanate. Bright green crystals of trans chlorobromodiethylenediamine cobaltic thiocyanate precipitate.

(a) $[Cl(H_2O)Co(C_2H_8N_2)_2]Br_2 \rightarrow [ClBrCo(C_2H_8N_2)_2]Br + H_2O$

(b) $[ClBrCo(C_2H_8N_2)_2]Br + NH_4SCN$
$\rightarrow [ClBrCo(C_2H_8N_2)_2]SCN + NH_4Br$

A. Werner, L. Gerb, S. Lorie and J. Rapiport, Ann., **386**, 119 (1912) *20*

$[CoH_2OClen_2]Br_2$

Δ **III-409**

$Na_2S_2O_6$

Chloroaquodiethylenediamine cobaltic bromide is heated to constant weight at 110°C, dissolved in cold water, filtered and treated with a solution of sodium dithionate. Trans chlorobromodiethylenediamine cobaltic dithionate is precipitated as small green crystals.

(a) $[Cl(H_2O)Co(C_2H_8N_2)_2]Br_2 \rightarrow [ClBrCo(C_2H_8N_2)_2]Br + H_2O$

(b) $2[ClBrCo(C_2H_8N_2)_2]Br + Na_2S_2O_6$
$\rightarrow [ClBrCo(C_2H_8N_2)_2]_2S_2O_6 + 2NaBr$

A. Werner, L. Gerb, S. Lorie and J. Rapiport, Ann., **386**, 119 (1912) *20*

$[Co(H_2O)_2en_2]Br_3$

$AgNO_3$ **III-410**

3 Grams of trans diaquodiethylenediamine cobaltic bromide are dissolved in the smallest volume of water possible at 25–30°C and an aqueous solution of silver nitrate added. The solution is filtered and the filtrate cooled in an ice bath. Brownish red crystals of trans diaquodiethylenediamine cobaltic nitrate deposit.

$$[(H_2O)_2Co(C_2H_8N_2)_2]Br_3 + 3AgNO_3 \rightarrow [(H_2O)_2Co(C_2H_8N_2)_2](NO_3)_3 + 3AgBr$$

A. Werner and K. Lange, Ann., **386**, 91 (1912) *20*

$[Co(H_2O)_2en_2]Br_3$

Ag_2SO_4 **III-411**

5 Grams of cis diaquodiethylenediamine cobaltic bromide are dissolved in water and treated with the calculated amount of silver sulphate. The solution is filtered and alcohol added to the filtrate until a permanent turbidity results. In from 1–2 days, red needles of cis diaquodiethylenediamine cobaltic sulfate deposit.

$$2[(H_2O)_2Co(C_2H_8N_2)_2]Br_3 + 3Ag_2SO_4 \rightarrow [(H_2O)_2Co(C_2H_8N_2)_2]_2(SO_4)_3 + 6AgBr$$

A. Werner and K. Lange, Ann., **386**, 89 (1912) *20*

$[CoNH_3H_2Oen_2]Br_3$

(HBr) **III-412**

Δ

2 Grams of trans aquoamminodiethylenediamino cobaltic bromide are dissolved in concentrated hydrobromic acid and the solution concentrated on the water bath. After a short time yellow crystals form. These are removed by filtration and the filtrate allowed to crystallize. Large dark reddish violet prismatic crystals of trans bromoamminodiethylenediamino cobaltic bromide with one H_2O deposit.

$$[H_2ONH_3Co(C_2H_8N_2)_2]Br_3 \rightarrow [BrNH_3Co(C_2H_8N_2)_2]Br_2 + H_2O$$

A. Werner and W. Boes, Ann., **386**, 184 (1912) *20*

$[Co(H_2O)_2en_2]Br_3$

HNO_3 **III-413**

5 Grams of cis diaquodiethylenediamino cobaltic bromide are dissolved in a small volume of water, the solution cooled in an ice bath and concentrated nitric acid added. Cis diaquodiethylenediamino cobaltic nitrate deposits and is recrystallized from water at 30°C slightly acidified with nitric acid. The compound is obtained as red crystals.

$$[(H_2O)_2Co(C_2H_8N_2)_2]Br_3 + 3HNO_3 \rightarrow [(H_2O)_2Co(C_2H_8N_2)_2](NO_3)_3 + 3HBr$$

A. Werner and K. Lange, Ann., **386**, 88 (1912) *20*

$[Co(H_2O)_2en_2]Br_3$

HNO_3 **III-414**

5 Grams of trans diaquodiethylenediamino cobaltic bromide are dissolved in the smallest volume of water possible and the solution cooled in an ice bath. Concentrated nitric acid is added until crystals begin to form. The solution is left in the ice bath for 30 minutes, the crystals removed and recrystallized from water acidified with nitric acid. Brownish red needles of trans diaquodiethylenediamino cobaltic nitrate are deposited.

$$[(H_2O)_2Co(C_2H_8N_2)_2]Br_3 + 3HNO_3 \rightarrow [(H_2O)_2Co(C_2H_8N_2)_2](NO_3)_3 + 3HBr$$

A. Werner and K. Lange, Ann., **386**, 91 (1912) *20*

$[Co(NH_3)H_2Oen_2]Br_3$

HNO_3 **III-415**

Concentrated nitric acid is added dropwise to a cooled saturated aqueous solution of trans aquoamminodiethylenediamino cobaltic bromide while stirring. A red crystalline precipitate forms. Addition of nitric acid is continued until about one third of the original volume of the solution has been added. Let the solution stand one hour and then cool in an ice-salt bath. Fiery red lustrous prismatic crystals of trans aquoamminodiethylenediamino cobaltic nitrate deposit.

$$[H_2ONH_3Co(C_2H_8N_2)_2]Br_3 + 3HNO_3 \rightarrow [H_2ONH_3Co(C_2H_8N_2)_2](NO_3)_3 + 3HBr$$

A. Werner and W. Boes, Ann., **386**, 190 (1912) *20*

$[Co(NH_3)H_2Oen_2]Br_3$

H_2PtCl_6 **III-416**

A few drops of acetic acid are added to a cooled saturated aqueous solution of trans aquoamminodiethylenediamino cobaltic bromide, then a solution of chloroplatinic acid is added. Trans aquoamminodiethylenediamino cobaltic chloroplatinate deposits as dark brownish red crystals which are soluble in water.

$$2[H_2ONH_3Co(C_2H_8N_2)_2]Br_3 + 3H_2PtCl_6 \rightarrow [H_2ONH_3Co(C_2H_8N_2)_2]_2(PtCl_6)_3 + 6HBr$$

A. Werner and W. Boes, Ann., **386**, 191 (1912) *20*

$[Co(H_2O)_2en_2]Br_3$

H_2SO_4 **III-417**

4 Grams of trans diaquodiethylenediamino cobaltic bromide are dissolved in a small volume of water and the solution cooled. Concentrated sulfuric acid is added slowly so that the temperature does not rise above 30°C. Alcohol is added to the solution and trans diaquodiethylenediamino cobaltic sulfate is obtained as a brownish red precipitate.

$$2[(H_2O)_2Co(C_2H_8N_2)_2]Br_3 + 3H_2SO_4 \rightarrow \underline{[(H_2O)_2Co(C_2H_8N_2)_2]_2(SO_4)_3} + 6HBr$$

A. Werner and K. Lange, Ann., **386**, 92 (1912) *20*

$[CoNH_3H_2Oen_2]Br_3$

KI **III-418**

Solid potassium iodide is added to a saturated aqueous solution of trans aquoamminodiethylenediamino cobaltic bromide until the solution is saturated. The reddish brown precipitate which forms is recrystallized from water by adding alcohol and concentrating quickly in vacuuo. This is done to prevent formation of the periodide. Trans aquoamminodiethylenediamino cobaltic iodide is obtained as brown, red, flat prismatic crystals with one H_2O. The salt is soluble in water.

$$[H_2ONH_3Co(C_2H_8N_2)_2]Br_3 + 3KI \rightarrow [H_2ONH_3Co(C_2H_8N_2)_2]I_3 + 3KBr$$

A. Werner and W. Boes, Ann., **386**, 190 (1912) *20*

$[Co(H_2O)_2en_2]Br_3$

KSCN **III-419**

The calculated amount of solid potassium thiocyanate is added to a concentrated aqueous solution of 5 grams of trans diaquodiethylenediamino cobaltic bromide and the solution triturated. The solution is cooled in an ice bath and dark brown crystals of trans diaquodiethylenediamino cobaltic thiocyanate with one half H_2O deposit.

$$[(H_2O)_2Co(C_2H_8N_2)_2]Br_3 + 3KSCN \rightarrow [(H_2O)_2Co(C_2H_8N_2)_2](SCN)_3 + 3KBr$$

A. Werner and K. Lange, Ann., **386**, 93 (1912) *20*

$[Co(NH_3)H_2Oen_2]Br_3$

K_2PtCl_4 **III-420**

An aqueous solution of trans aquoamminodiethylenediamino cobaltic bromide is treated with a solution of potassium chloroplatinite and small bright brown crystals of trans aquoamminodiethylenediamino cobaltic chloroplatinite with two H_2O deposit.

$$2[H_2ONH_3Co(C_2H_8N_2)_2]Br_3 + 3K_2PtCl_4 \rightarrow [H_2ONH_3Co(C_2H_8N_2)_2]_2(PtCl_4)_3 + 6KBr$$

A. Werner and W. Boes, Ann., **386**, 191 (1912) *20*

$[Coen_3]Br_3$

$K_3[Cr(C_2O_4)_3]$ **III-421**

A dilute aqueous solution of triethylenediamino cobaltic bromide is added to one of chromium potassium oxalate and a brown crystalline precipitate of triethylenediaminocobaltictrioxalatochromate with six H_2O is formed.

$$[Co(C_2H_4(NH_2)_2)_3]Br_3 + (Cr(C_2O_4)_3)K_3 \rightarrow [Co(C_2H_4(NH_2)_2)_3]\cdot[Cr(C_2O_4)_3] + 3KBr$$

P. Pfeiffer, Ann., **346**, 54 (1906) *20*

$[CoNH_3H_2Oen_2]Br_3$

$NaNO_2$ **III-422**

A solution of cis aquoamminodiethylenediamino cobaltic bromide, saturated at 25°C, is treated with an excess of a saturated aqueous solution of sodium nitrite, a few drops of acetic acid added and the solution warmed to 40°C. Ammonia is evolved if the solution is heated too hot. The color of the solution changes from cherry red to orange yellow. The solution is cooled and allowed to stand 24 hours when an orange yellow precipitate forms. It is dissolved in water, warmed to 35°C and potassium bromide added. As the solution cools, cis nitritoamminodiethylenediamino cobaltic bromide deposits in almost pure form. It may be recrystallized from warm water and is obtained as large dark yellow tablets.

$$[H_2ONH_3Co(C_2H_8N_2)_2]Br_3 + NaNO_2 \rightarrow [NO_2NH_3Co(C_2H_8N_2)_2]Br_2 + NaBr + H_2O$$

A. Werner and W. Boes, Ann., **386**, 218 (1912) *20*

$[Co(H_2O)_2en_2]Br_3$

$Na_2S_2O_6$ **III-423**

4 Grams of trans diaquodiethylenediamino cobaltic bromide are dissolved in a small volume of water and treated with the calculated amount of sodium dithionate in solution. The solution is cooled in an ice bath and ethyl alcohol added. The precipitated trans diaquodiethylenediamino cobaltic dithionate is recrystallized from water by the addition of alcohol. The compound is obtained as brownish red needles.

$$2[(H_2O)_2Co(C_2H_8N_2)_2]Br_3 + 3Na_2S_2O_6 \rightarrow [(H_2O)_2Co(C_2H_8N_2)_2]_2(S_2O_6)_3 + 6NaBr$$

A. Werner and K. Lange, Ann., **386**, 92 (1912) *20*

$[Co(H_2O)_2en_2]Br_3$

Δ **III-424**

5 Grams of cis diaquodiethylenediamino cobaltic bromide are shaken with a small volume of water so that not all of the salt dissolves. The solution is heated on the water bath to 40°C. When all of the salt dissolves, the color of the solution changes from red to violet. The solution is now cooled and solid sodium bromide added to saturation. Cis bromoaquodiethylenediamino cobaltic bromide is precipitated as a fine violet crystalline mass which may be crystallized from water by the addition of sodium bromide.

$$[(H_2O)_2Co(C_2H_8N_2)_2]Br_3 + \text{heat} \rightarrow [BrH_2OCo(C_2H_8N_2)_2]Br_2 + H_2O$$

A. Werner and R. Schmidt, Ann., **386**, 128 (1912) *20*

$[CoC_3H_2O_4en_2]C_3H_3O_4$

KNO_3 **III-425**

An aqueous solution of malonatodiethylenediamine cobaltic acid malonate is treated with solid potassium nitrate and the precipitate which forms is recrystallized from warm water. Small red lustrous leaflets of malonatodiethylenediamino cobaltic nitrate are obtained. They are soluble in water.

$$[(C_3H_2O_4)Co(C_2H_8N_2)_2](C_3H_3O_4) + KNO_3 \rightarrow [(C_3H_2O_4)Co(C_2H_8N_2)_2]NO_3 + C_3H_3O_4K$$

A. Werner and J. Rapiport, Ann., **386**, 80 (1912) *20*

$[CoC_3H_2O_4en_2]C_3H_3O_4$

NH_4SCN **III-426**

Malonatodiethylenediamino cobaltic acid malonate is dissolved in water, the solution cooled, filtered and solid ammonium thiocyanate added to the filtrate. After a short time a red precipitate forms. Concentration of the solution and cooling produces red lustrous leaflets of malonatodiethylenediamino cobaltic thiocyanate.

$$[(C_3H_2O_4)Co(C_2H_8N_2)_2](C_3H_3O_4) + NH_4SCN \rightarrow [(C_3H_2O_4)Co(C_2H_8N_2)_2]SCN + C_3H_3O_4NH_4$$

A. Werner and J. Rapiport, Ann., **386**, 80 (1912) *20*

$[CoCl_2en_2]Cl$

$AgNO_3$ **III-427**

6 Grams of neutral trans dichlorodiethylenediamino cobaltic chloride are added to 3 cc of water and 2.5 cc of not too concentrated nitric acid, and the mixture shaken. A concentrated aqueous solution of 7.2 grams of silver nitrate is added and the solution warmed slightly until all of the green salt has dissolved, the solution becomes bordeaux red and silver chloride separates. Solid sodium bromide is now added to precipitate the excess silver. The solution is now filtered and the filtrate saturated with solid sodium bromide. A violet crystalline powder separates. The solution is allowed to stand one hour and the precipitate removed. This precipitate is cis bromoaquodiethylenediamino cobaltic bromide.

(a) $$[Cl_2Co(C_2H_8N_2)_2]Cl + 3AgNO_3 + H_2O \rightarrow [NO_3(H_2O)Co(C_2H_8N_2)_2](NO_3)_2 + 3AgCl$$

(b) $$[NO_3(H_2O)Co(C_2H_8N_2)_2](NO_3)_2 + 3NaBr \rightarrow [Br(H_2O)Co(C_2H_8N_2)_2]Br_2 + 3NaNO_3$$

A. Werner and R. Schmidt, Ann., **386**, 126 (1912) *20*

$[CoCO_3en_2]Cl$

$AgNO_3$ **III-428**

The calculated quantity of silver nitrate in solution is added to a hot solution of 5 grams of carbonatodiethylenediamino cobaltic chloride. The solution is filtered and concentrated on the water bath. Carbonatodiethylenediamino cobaltic nitrate crystallizes in dark, brownish-red lustrous needles with one H_2O. 1 gram dissolves in 20 cc of water at 60°C.

$$[CO_3Co(C_2H_8N_2)_2]Cl + AgNO_3 \rightarrow [CO_3Co(C_2H_8N_2)_2]NO_3 + (AgCl)$$

A. Werner and J. Rapiport, Ann., **386**, 75 (1912) *20*

$[CoCl_2en_2]Cl$

$CH_2(COOH)_2$ **III-429**

When a cold concentrated solution of dichlorodiethylenediamino cobaltic chloride is mixed with equimolar proportions of malonic acid, an addition compound is precipitated.

$$[Cl_2Co(NH_2C_2H_4NH_2)_2]Cl + 2CH_2(COOH)_2 + 2H_2O$$
$$\rightarrow \underline{[Cl_2Co(NH_2C_2H_4NH_2)_2]OCOCH_2COOH \cdot CH_2(COOH)_2 \cdot 2H_2O}$$
$$+ (HCl)$$

T. S. Price and S. A. Brazier, J. Chem. Soc. (London), **107**, 1728 (1915) *57*

$[Cl_2Co(C_2H_8N_2)_2]Cl$

C_2H_7N **III-430**

5.7 Grams of trans dichlorodiethylenediamino cobaltic chloride are added to 5 cc of a 20% aqueous solution of ethylamine and strongly heated. Rhombic crystals soon begin to separate. The solution is allowed to stand in an ice bath for some time, the crystals filtered off and washed with dilute hydrochloric acid. Deep ruby red crystals of monochlorodiethylenediaminoethylamino cobaltic chloride with one H_2O are formed. The yield is 4.5 grams and the compound is easily soluble in water. Aqueous solutions are stable when boiled.

$$[Cl_2Co(C_2H_8N_2)_2]Cl + C_2H_7N \rightarrow [ClCo(C_2H_8N_2)_2(C_2H_7N)]Cl_2$$

J. Meisenheimer and E. Kinderlen, Ann., **438**, 254 (1924) *20*

$[Cl_2Co(C_2H_8N_2)_2]Cl$

C_3H_7N **III-431**

Trans dichlorodiethylenediamino cobaltic chloride is added to twice its weight of water and one molecular weight of allylamine added. The mixture is shaken and the chloro compound soon dissolves while crystals begin to form. After some time the crystalline mass is filtered and the residue obtained is chlorodiethylenediaminoallylamino cobaltic chloride. The compound is obtained as violet red rhombic plates.

$$[Cl_2Co(C_2H_8N_2)_2]Cl + C_3H_7N \rightarrow [ClCo(C_2H_8N_2)_2(C_3H_7N)]Cl_2$$

J. Meisenheimer and E. Kinderlen, Ann., **438**, 248 (1924) *20*

$[Cl_2Co(C_2H_8N_2)_2]Cl$

$C_3H_7O_2N$

NaOH **III-432**

29 Grams of trans dichlorodiethylenediamino cobaltic chloride are added during fifteen minutes to a hot aqueous solution of 9 grams of sarcosine (methylaminoacetic acid) and 4 grams of sodium hydroxide in 30 cc of water. Yellow brown prisms begin to deposit from the deep red colored solution. The solution is cooled and allowed to stand for six hours, then filtered to remove the crystals of the sodium chloride double salt of triethylenediamine cobaltic chloride. 40–50 cc of absolute alcohol is added to the filtrate and 14–15 grams of sarcosine diethylenediamino cobaltic chloride precipitates as strawberry red plates. An additional 3–4 grams may be obtained by the further addition of alcohol. The compound is recrystallized from an equal weight of water by the addition of alcohol. The compound crystallizes with one and one half H_2O.

$$[Cl_2Co(C_2H_8N_2)_2]Cl + C_3H_7O_2N + NaOH \rightarrow [(C_3H_6O_2N)Co(C_2H_8N_2)_2]Cl_2 + H_2O + NaCl$$

J. Meisenheimer and E. Kinderlen, Ann., **438**, 270 (1924) *20*

$[Cl_2Co(C_2H_8N_2)_2]Cl$

$C_6H_5NH_2$ **III-433**

28.6 Grams of acid free trans dichlorodiethylenediamino cobaltic chloride are suspended in 40 cc of water and 9.3 gm. of aniline added. The solution is shaken in a shaking machine and the color changes to a dirty red. After 2–3 hours the first crystals begin to form. Shaking is continued for 24 hours and the crystalline mass removed by filtration. The crystals are washed with 50% alcohol, 95% alcohol and finally ether. The yield is 33–35 grams of monochlorodiethylenediaminoanilino cobaltic chloride which is obtained as reddish violet prisms with one H_2O. The compound may be recrystallized by dissolving 5 grams in 18 cc of water at 70°C, filtering and adding 20 cc of 23% hydrochloric acid at 80–90°C to the residue, combining the two solutions and allowing to crystallize. The compound is insoluble in alcohol, fairly soluble in dilute hydrochloric acid and more soluble in concentrated acid. The odor of aniline can be detected in aqueous solutions warmed to 70°C and sodium hydroxide solutions decompose the compound immediately. The compound may be boiled with hydrochloric acid for a short time without decomposition.

$$[Cl_2Co(C_2H_8N_2)_2]Cl + C_6H_5NH_2 \rightarrow [ClCo(C_2H_8N_2)_2(C_6H_5NH_2)]Cl_2$$

J. Meisenheimer and E. Kinderlen, Ann., **438**, 242 (1924) *20*

$[Cl_2Co(C_2H_8N_2)_2]Cl$

C_7H_9N **III-434**

A suspension of 14.5 grams of acid free trans dichlorodiethylenediamino cobaltic chloride in 25 cc of water is shaken with one of 5.5 grams of p-toluidine in 20 cc of alcohol. The chloride goes into solution and soon chlorodiethylenediamino-p-toluidino cobaltic chloride begins to deposit. At the end of one hour the solution has become a semi-solid mass. Allow to stand overnight, filter and wash with dilute hydrochloric acid, alcohol and then ether. Yield 18 grams. The compound is purified by dissolving 6 grams in 90 cc of water at 40°C, the residue dissolved in 60 cc of 35% hydrochloric acid and the solutions combined. The compound crystallizes as brownish red leaves with one H_2O.

$$[Cl_2Co(C_2H_8N_2)_2]Cl + C_7H_9N \rightarrow [ClCo(C_2H_8N_2)_2(C_7H_9N)]Cl_2$$

J. Meisenheimer and E. Kinderlen, Ann., **438**, 246 (1924) *20*

$[Cl_2Co(C_2H_8N_2)_2]Cl$

C_7H_9N **III-435**

5.7 Grams of trans dichlorodiethylenediamino cobaltic chloride together with 2.2 grams of benzyl amine are added to 13 cc of water and shaken. The solution becomes violet red in color and soon a thick crystalline mass separates. The solution is cooled in ice and the residue removed by filtration. 5 grams of chlorodiethylenediamino-benzylamino cobaltic chloride are obtained as violet rose colored needles which contain one H_2O. The compound may be recrystallized from hot hydrochloric acid.

$$[Cl_2Co(C_2H_8N_2)_2]Cl + C_7H_9N \rightarrow [ClCo(C_2H_8N_2)_2(C_7H_9N)]Cl_2$$

J. Meisenheimer and E. Kinderlen, Ann., **438**, 250 (1924) *20*

$[Co(SCN)_2en_2]Cl$

Cl_2 **III-436**

Chlorine is passed into a cold aqueous solution of dithiocyanodiethylenediamino cobalt monochloride. The solution changes to a fiery red color and the temperature rises as the reaction proceeds. The solution is concentrated to one fourth its volume and, as it cools, crystals form. These are dissolved in water, acidified with hydrochloric acid and absolute alcohol added until turbidity results. Crystals of diamminodiethylenediaminocobalt trichloride with one H_2O separate. They are bright yellow.

$$[Co(SCN)_2(C_2H_8N_2)_2]Cl + 8Cl_2 + 12H_2O \rightarrow [Co(NH_3)_2(C_2H_8N_2)_2]Cl_3 + 2H_2SO_4 + 14HCl + 2CO_2$$

A. Werner, F. Braunlich and E. Rogowina, Ann., **351**, 71 (1907) *20*

$[CoNO_2NCSen_2]Cl$

Cl_2 **III-437**

HNO_3

Chlorine gas is passed into an aqueous solution of trans nitroisothiocyanatodiethylenediamino cobaltic chloride and the reddish brown color of the solution changes to wine red in a few minutes. The solution becomes warm during the reaction. Nitric acid is added to the solution and orange red crystals of trans chloronitrodiethylenediamino cobaltic nitrate are deposited.

$$[NO_2NCSCo(C_2H_8N_2)_2]Cl + HNO_3 \rightarrow [ClNO_2Co(C_2H_8N_2)_2]NO_3 + HSCN$$

A. Werner and N. Gosling, Ann., **386**, 246 (1912) *20*

$[CoCO_3en_2]Cl$

HBr **III-438**

Carbonatodiethylenediamino cobaltic chloride is finely pulverized and added, in small portions, to 5 grams of very cold concentrated hydrobromic acid until the reaction product is gray green in color (not bright green). Absolute alcohol is added and, after ten minutes, the material is filtered. The residue is rubbed on a porous plate and then with a very small volume of ice cold water. A portion of the residue dissolves to form a dark red solution. The residue is removed by filtration and the extraction continued until the extract is no longer red but a gray violet color. Solid sodium bromide is added to the red extract and red crystals of cis dibromodiethylenediamino cobaltic bromide deposit.

$$[CO_3Co(C_2H_8N_2)_2]Cl + 3HBr \rightarrow [Br_2Co(C_2H_8N_2)_2]Br + HCl + H_2O + CO_2$$

A. Werner, L. Gerb, S. Lorie and J. Rapiport, Ann., **386**, 114 (1912) *20*

$[CoCO_3en_2]Cl$

HBr **III-439**

Carbonatodiethylenediamino cobaltic chloride is added to concentrated hydrobromic acid (sp.gr. 1.4) and the mixture shaken. Carbon dioxide is evolved and a bluish violet crystalline mass precipitates. This precipitate is a mixture of the easily soluble violet cis bromoaquodiethylenediamino cobaltic bromide and the difficultly soluble gray violet cis dibromodiethylenediamino cobaltic bromide. These salts are separated by fractional solution as the violet salt (bromoaquo salt) is easily soluble and may be dissolved from the less soluble gray violet dibromo salt. Solid sodium bromide precipitates cis bromoaquodiethylenediamino cobaltic bromide.

$$2[CO_3Co(C_2H_8N_2)_2]Cl + 6HBr \rightarrow [Br_2Co(C_2H_8N_2)_2]Br + [BrH_2OCo(C_2H_8N_2)_2]Br_2 + 2CO_2 + 2HCl + H_2O$$

A. Werner and R. Schmidt, Ann., **386**, 127 (1912) *20*

$[CoClNCSen_2]Cl$

HBr **III-440**

Cis chloroisothiocyanatodiethylenediamino cobaltic chloride is added to concentrated hydrobromic acid and the mixture triturated. The mixture is warmed slightly and slowly, then allowed to cool. Dark red crystals of cis chloroisothiocyanatodiethylenediamino cobaltic bromide with one and one half H_2O deposit.

$$[ClNCSCo(C_2H_8N_2)_2]Cl + HBr \rightarrow [ClNCSCo(C_2H_8N_2)_2]Br + HCl$$

A. Werner and R. Schmidt, Ann., **386**, 142 (1912) *20*

$[Co(NH_3)_2Cl_2en]Cl$

HBr **III-441**

A concentrated aqueous solution of trans dichloroethylenediaminodiammino cobaltic chloride is treated with hydrobromic acid. The solution is stirred and a heavy green precipitate forms. Hydrobromic acid is added until no more precipitate forms. It is removed and dried on a porous plate. The compound is trans dichloroethylenediaminodiammino cobaltic bromide.

$$[Cl_2Co(C_2H_8N_2)(NH_3)_2]Cl + HBr \rightarrow [Cl_2Co(C_2H_8N_2)(NH_3)_2]Br + HCl$$

A. Werner and F. Chaussy, Ann., **386**, 259 (1912) *20*

$[CoCl_2en_2]Cl$

HCl **III-442**

When hydrochloric acid is added to a solution of dichlorodiethylenediamino cobaltic chloride, an addition compound is precipitated.

$$[Cl_2Co(NH_2C_2H_4NH_2)_2]Cl + HCl + 2H_2O \rightarrow \underline{[Cl_2Co(NH_2C_2H_4NH_2)_2]Cl \cdot HCl \cdot 2H_2O}$$

T. S. Price and S. A. Brazier, J. Chem. Soc. (London), **107**, 1716 (1915) *57*

$[CoCO_3en_2]Cl$

HCl **III-443**

Hydrochloric acid is saturated with hydrochloric acid gas at 0°C and frozen in liquid air. The crystals which freeze out are used as solvent. In this is dissolved carbonatodiethylenediamino cobaltic chloride. The temperature of this solution is allowed to rise to room temperature, and the color of the solution changes from red to violet. Alcohol is added to the solution and dark violet crystals of cis dichlorodiethylenediamino cobaltic chloride form.

$$[CO_3Co(C_2H_8N_2)_2]Cl + 2HCl \rightarrow [Cl_2Co(C_2H_8N_2)_2]Cl + CO_2 + H_2O$$

A. Werner and R. Bosshard, Ann., **386**, 108 (1912) *20*

$[Co(NO_2)_2pn_2]Cl$

HCl **III-444**

Trans dinitroditrimethylenediamino cobaltic chloride is added to concentrated hydrochloric acid. Gas is evolved and a green solution results from which green crystals are deposited. The compound is trans dichloroditrimethylenediamino cobaltic chloride. It is recrystallized from water acidified with hydrochloric acid. The salt dissolves in water to form a green solution which changes to violet. A solution of 1 gram in 20 cc of water reacts as follows: Concentrated hydrochloric acid gives a violet solution which changes to green and green needles are deposited. Concentrated hydrobromic acid produces a green solution. Concentrated potassium hydroxide and ammonium hydroxide produce a red solution. Potassium iodide gives a thick brown precipitate. Potassium bromide gives a grass green precipitate. Potassium thiocyanate gives a red brown solution from which reddish brown crystals separate. Potassium permanganate gives dark brown violet crystals.

$$[(NO_2)_2Co(C_3H_{10}N_2)_2]Cl + 2HCl \rightarrow [Cl_2Co(C_3H_{10}N_2)_2]Cl + 2HNO_2$$

A. Werner and G. Lindenberg, Ann., **386**, 270 (1912) *20*

$[Co(NH_3)_2CO_3en]Cl$

HCl **III-445**

NH_4Br

Carbonatoethylenediaminodiammino cobaltic chloride is treated with concentrated hydrochloric acid which has been saturated with hydrogen chloride gas at −16°C. The carbonato salt dissolves and solid ammonium bromide is added. This reaction produces a red solution which changes to violet and a bluish violet precipitate of cis dichloroethylenediaminodiammino cobaltic bromide forms. This is dissolved in water and reprecipitated by the addition of ammonium bromide.

(a) $[CO_3Co(C_2H_8N_2)(NH_3)_2]Cl + 2HCl \rightarrow [Cl_2Co(C_2H_8N_2)(NH_3)_2]Cl + H_2O + CO_2$

(b) $[Cl_2Co(C_2H_8N_2)(NH_3)_2]Cl + NH_4Br \rightarrow [Cl_2Co(C_2H_8N_2)(NH_3)_2]Br + NH_4Cl$

A. Werner and F. Chaussy, Ann., **386**, 262 (1912) *20*

$[Co(NH_3)_2Cl_2en]Cl$

HNO_3 **III-446**

1 Gram of trans dichloroethylenediaminodiammino cobaltic chloride is dissolved in the minimum amount of water, filtered, and nitric acid (1:1) added dropwise. A green precipitate forms which is removed by filtration, washed with water and dried on a porous plate. The salt formed is trans dichloroethylenediaminodiammino cobaltic nitrate.

$[Cl_2Co(C_2H_8N_2)(NH_3)_2]Cl + HNO_3 \rightarrow [Cl_2Co(C_2H_8N_2)(NH_3)_2]NO_3 + HCl$

A. Werner and F. Chaussy, Ann., **386**, 258 (1912) *20*

$[CoCl_2en_2]Cl$

$H_2C_2O_4$ **III-447**

When a cold concentrated solution of dichlorodiethylenediamino cobaltic chloride is mixed with an equimolar quantity amount of oxalic acid, an emerald-green addition compound is precipitated. The same precipitate is obtained with ammonium acid oxalate and potassium acid oxalate.

$$[Cl_2Co(NH_2C_2H_4NH_2)_2]Cl + H_2C_2O_4 + 2H_2O \rightarrow \underline{[Cl_2Co(NH_2C_2H_4NH_2)_2]C_2O_4H \cdot 2H_2O} + (HCl)$$

T. S. Price and S. A. Brazier, J. Chem. Soc. (London), **107**, 1926 (1915) *57*

$[Cl_2Co(C_2H_8N_2)_2]Cl$

H_2O **III-448**

2.8 Grams of trans dichlorodiethylenediamino cobaltic chloride are dissolved in ice water and 0.8 gram of diethylamine together with 3 grams of ice added. Partial solution results and the color of the solution changes from green to blue green. After 5 minutes solution is complete and it sets to a semisolid crystalline mass. The solution is filtered and the crystalline residue washed with 10 cc of 70% alcohol then with 95% alcohol and finally ether. 2.0 grams of trans chlorohydroxodiethlenediamino cobaltic chloride are obtained as grey crystals.

$$[Cl_2Co(C_2H_8N_2)_2]Cl + H_2O \rightarrow [ClCo(C_2H_8N_2)_2OH]Cl + HCl$$

J. Meisenheimer and E. Kinderlen, Ann., **438**, 255 (1924) *20*

$[CoClNCSen_2]Cl$

H_2O_2

HCl **III-449**

Trans chloroisothiocyanatodiethylenediamino cobaltic chloride may be oxidized with hydrogen peroxide in the presence of hydrochloric acid to form trans chloroamminodiethylenediamino cobaltic chloride.

$$[ClNCSCo(C_2H_8N_2)_2]Cl + 4H_2O_2 + HCl \rightarrow [ClNH_3Co(C_2H_8N_2)_2]Cl_2 + CO_2 + H_2SO_4 + 2H_2O$$

A. Werner and R. Schmidt, Ann., **386**, 169 (1912) *20*

$[CoCl_2pn_2]Cl$

H_2PtCl_6 **III-450**

1 Gram of trans dichloroditrimethylenediamino cobaltic chloride is dissolved in 30–35 cc of water, 10–15 drops of concentrated hydrochloric acid and 20–25 drops of an aqueous solution of chloroplatinic acid added. The solution is allowed to stand overnight and green crystals of trans dichloroditrimethylenediamino cobaltic chloroplatinate deposit.

$$2[Cl_2Co(C_3H_{10}N_2)_2]Cl + H_2PtCl_6 \rightarrow [Cl_2Co(C_3H_{10}N_2)_2]_2 \cdot PtCl_6 + 2HCl$$

A. Werner and G. Lindenberg, Ann., **386**, 271 (1912) *20*

$[Co(NH_3)_2Cl_2en]Cl$

H_2SO_4 **III-451**

A concentrated aqueous solution of trans dichloroethylenediaminodiammino cobaltic chloride is cooled to 4°C in an ice salt bath and dilute sulfuric acid (1:1) added dropwise while stirring. After about 10 minutes, a turbidity develops and the solution is then allowed to stand for some time at a low temperature. A greenish precipitate of trans dichloroethylenediaminodiammino cobaltic bisulfate with one H_2O forms.

$$[Cl_2Co(C_2H_8N_2)(NH_3)_2]Cl + H_2SO_4 \rightarrow [Cl_2Co(C_2H_8N_2)(NH_3)_2] \cdot HSO_4 + HCl$$

A. Werner and F. Chaussy, Ann., **386**, 260 (1912) *20*

$[CoCl_2en_2]Cl$

H_2SO_4 **III-452**

5 Grams of trans dichlorodiethylenediamino cobaltic chloride are dissolved in 15 cc of water and concentrated sulphuric acid added dropwise. A green precipitate forms which is recrystallized from water acidified with sulfuric acid and trans dichlorodiethylenediamino cobaltic hydrogensulfate is deposited.

$$[Cl_2Co(C_2H_8N_2)_2]Cl + H_2SO_4 \rightarrow [Cl_2Co(C_2H_8N_2)_2] \cdot HSO_4 + HCl$$

A. Werner and R. Bosshard, Ann., **386**, 106 (1912) *20*

$[CoSO_3en_2]Cl$

$KAuCl_4$ **III-453**

A concentrated aqueous solution of sulfitodiethylenediamino cobaltic chloride is treated with a solution of potassium chloroaurate and fine yellowish-brown scales of sulfitodiethylenediamino cobaltic chloroaurate with three H_2O form.

$$[SO_3Co(C_2H_8N_2)_2]Cl + KAuCl_4 \rightarrow [SO_3Co(C_2H_8N_2)_2] \cdot AuCl_4 + KCl$$

A. Werner and M. Pokrowska, Ann., **386**, 85 (1912) *20*

$[ClCo(C_2H_8N_2)_2OH]Cl$

KBr **III-454**

3 Grams of chlorodiethylenediaminohydroxo cobaltic chloride are dissolved in 15 cc of water, faintly warmed and 3 grams of solid potassium bromide added. Hydroxoaquodiethylenediamino cobaltic bromide precipitates as a red precipitate which is washed with a little water and then recrystallized from aqueous solution by the addition of alcohol. Yield 2 grams.

$$[ClCo(C_2H_8N_2)_2OH]Cl + 2KBr + H_2O \rightarrow [H_2OCo(C_2H_8N_2)_2OH]Br_2 + 2KCl$$

J. Meisenheimer and E. Kinderlen, Ann., **438**, 257 (1924) *20*

$[CoCO_3en_2]Cl$

KBr **III-455**

5 Grams of carbonato diethylenediamino cobaltic chloride is dissolved in 150 cc of water and treated with solid potassium bromide. Brownish red carbonato diethylenediamino cobaltic bromide precipitates. It is recrystallized from warm water. Both anhydrous and hydrous crystals are obtained. The hydrous form is dark red hexagonal prisms. 1 gram dissolves in 30 cc of water at 50°C.

$$[CO_3Co(C_2H_8N_2)_2]Cl + 2KBr \rightarrow [CO_3Co(C_2H_8N_2)_2]Br + (2KCl)$$

A. Werner and J. Rapiport, Ann., **386**, 73 (1912) *20*

$[Co(NCS)_2en_2]Cl$

KBr **III-456**

2 Grams of cis diisothiocyanatodiethylenediamino cobaltic chloride are dissolved in 100 cc of water by heating. A few drops of acetic acid are added and the solution cooled. Solid potassium bromide is added in excess and bright red crystals are precipitated. These are dissolved in hot water acidified with acetic acid, an equal volume of water added and the solution concentrated over sulphuric acid. Cis diisothiocyanatodiethylenediamino cobaltic bromide is deposited as red leafy crystals or needles with one half H_2O.

$$[(NCS)_2Co(C_2H_8N_2)_2]Cl + KBr \rightarrow [(NCS)_2Co(C_2H_8N_2)_2]Br + KCl$$

A. Werner and C. Rix, Ann., **386**, 196 (1912) *20*

$[CoNO_2NCSen_2]Cl$

KBr **III-457**

3 Grams of cis nitritoisothiocyanatodiethylenediamino cobaltic chloride are dissolved in 70 cc of water containing acetic acid and potassium bromide added in excess. Cis nitritoisothiocyanatodiethylenediamino cobaltic bromide deposits as a brownish red crystalline precipitate which is recrystallized from water acidified with acetic acid and bright brown crystals deposit.

$$[NO_2NCSCo(C_2H_8N_2)_2]Cl + KBr \rightarrow [NO_2NCSCo(C_2H_8N_2)_2]Br + KCl$$

A. Werner and C. Rix, Ann., **386**, 234 (1912) *20*

$[CoNO_2NCSen_2]Cl$

KBr **III-458**

A solution of 3 grams of trans nitritoisothiocyanatodiethylenediamino cobaltic chloride in 24 cc of water is treated with solid potassium bromide and a brown crystalline precipitate forms. This is removed by suction, washed with alcohol and ether then recrystallized from hot water. Large brown tablets of trans nitritoisothiocyanatodiethylenediamino cobaltic bromide with one H_2O deposit.

$$[NO_2NCSCo(C_2H_8N_2)_2]Cl + KBr \rightarrow [NO_2NCSCo(C_2H_8N_2)_2]Br + KCl$$

A. Werner and N. Gosling, Ann., **386**, 244 (1912) *20*

$[CoCO_3en_2]Cl$

KCNS **III-459**

To a concentrated water solution of carbonatodiethylenediamino cobaltic chloride solid potassium thiocyanate is added. The corresponding thiocyanate crystallizes as a purple-red powder.

$$[CO_3Co(C_2H_8N_2)_2]Cl + KCNS \rightarrow [CO_3Co(C_2H_8N_2)_2]CNS + KCl$$

A. Werner, Ann., **386**, 75 (1912) *41* *20*

$[CoC_2O_4en_2]Cl$

KI **III-460**

4 Grams of oxalato diethylenediamino cobaltic chloride are dissolved in 15 cc of water saturated at 0°C with ammonia. The solution is heated for two hours in a bomb at 110°C. A saturated aqueous solution of potassium iodide is added to this solution and red plates of oxalato-diethylenediamino cobaltic iodide deposit.

$$[C_2O_4Co(C_2H_8N_2)_2]Cl + KI \rightarrow [C_2O_4Co(C_2H_8N_2)_2]I + KCl$$

A. Werner and J. Rapiport, Ann., **386**, 77 (1912) *20*

$[CoCO_3en_2]Cl$

KI **III-461**

5 Grams of carbonatodiethylenediamino cobaltic chloride is dissolved in 200 cc of water and solid potassium iodide added. A bluish-red crystalline powder precipitates. This is recrystallized from hot water and lustrous, dark red prisms of carbonatodiethylenediamino cobaltic iodide are obtained. One gram of the compound dissolves in 70 cc of water at 80°C.

$$[CO_3Co(C_2H_8N_2)_2]Cl + KI \rightarrow [CO_3Co(C_2H_8N_2)_2]I + (KCl)$$

A. Werner and J. Rapiport, Ann., **386**, 74 (1912) *20* *41*

$[Co(NCS)_2en_2]Cl$

KI **III-462**

3 Grams of cis diisothiocyanatodiethylenediamino cobaltic chloride are dissolved in hot water slightly acidified with acetic acid. Solid potassium iodide is added and a red crystalline precipitate forms which is filtered off, dissolved in hot water, diluted with an equal volume of water and allowed to concentrate over sulphuric acid. Cis diisothiocyanatodiethylenediammino cobaltic iodide is deposited as lustrous red needles which are difficulty soluble in cold but easily soluble in hot water.

$$[(NCS)_2Co(C_2H_8N_2)_2]Cl + KI \rightarrow [(NCS)_2Co(C_2H_8N_2)_2]I + KCl$$

A. Werner and C. Rix., Ann., **386**, 197 (1912)

20

$[Co(NH_3)_2Cl_2en]Cl$

KI **III-463**

Solid potassium iodide is added to a cold filtered aqueous solution of trans dichloroethylenediamino diammino cobaltic chloride until it is saturated and a brown precipitate of trans dichloroethylenediamino-diammino cobaltic iodide forms. It is filtered off and dried on a porous plate.

$$[Cl_2Co(C_2H_8N_2)(NH_3)_2]Cl + KI \rightarrow [Cl_2Co(C_2H_8N_2)(NH_3)_2]I + KCl$$

A. Werner and F. Chaussy, Ann., **386**, 259 (1912)

20

$[CoNO_2NCSen_2]Cl$

KI **III-464**

3 Grams of trans nitritoisothiocyanatodiethylenediamino cobaltic chloride are dissolved in 24 cc of water and solid potassium iodide added. A brown precipitate forms which is recrystallized from hot water. Brown crystals of trans nitritoisothiocyanatodiethylene-diamino cobaltic iodide are formed.

$$[NO_2NCSCo(C_2H_8N_2)_2]Cl + KI \rightarrow [NO_2NCSCo(C_2H_8N_2)_2]I + KCl$$

A. Werner and N. Gosling, Ann., **386**, 244 (1912)

20

$[CoNO_2NCSen_2]Cl$

KI **III-465**

3 Grams of cis nitritoisothiocyanatodiethylenediamino cobaltic chloride are added to 70 cc of water acidified with acetic acid and the solution heated on the water bath until solution is complete. Solid potassium iodide is added while stirring and cis nitritoisothiocyanatodiethylenediamino cobaltic iodide precipitates as a brownish red crystalline powder. The solution is concentrated over sulphuric acid and the compound is obtained as brown prisms.

$$[NO_2NCSCo(C_2H_8N_2)_2]Cl + KI \rightarrow [NO_2NCSCo(C_2H_8N_2)_2]I + KCl$$

A. Werner and C. Rix, Ann., **386**, 235 (1912) *20*

$[Co(NH_3)_4(NO_2)_2]Cl$

$K[(NH_3)_2Co(NO_2)_4]$ **III-466**

A solution of croceo cobalt chloride will react with a solution of potassium diammino cobaltic nitrite yielding a yellow precipitate.

$$[(NO_2)_2 \cdot Co(NH_3)_4]Cl + K[(NH_3)_2 \cdot Co(NO_2)_4]$$
$$\rightarrow \underline{[(NO_2)_2Co(NH_3)_4][(NO_2)_2(NH_3)_2Co(NO_2)_2]} + KCl$$

S. M. Jörgensen, Z. anorg. Chem., **5**, 147 (1894) *28*

$[Co(NCS)_2en_2]Cl$

KNO_3 **III-467**

2 Grams of cis diisothiocyanatodiethylenediamino cobaltic chloride are dissolved in water acidified with acetic acid and solid potassium nitrate added. Cis diisothiocyanatodiethylenediamino cobaltic nitrate is precipitated as small red prisms.

$$[(NCS)_2Co(C_2H_8N_2)_2]Cl + KNO_3$$
$$\rightarrow [(NCS)_2Co(C_2H_8N_2)_2]NO_3 + KCl$$

A. Werner and C. Rix, Ann., **386**, 198 (1912) *20*

$[CoNO_2NCSen_2]Cl$

KNO_3 **III-468**

3 Grams of trans nitritoisothiocyanatodiethylenediamino cobaltic chloride are dissolved in 24 cc of water and solid potassium nitrate added. A yellowish brown precipitate forms which is recrystallized from hot water and brown tablets of trans nitritoisothiocyanatodiethylenediamino cobaltic nitrate with one H_2O are deposited.

$$[NO_2NCSCo(C_2H_8N_2)_2]Cl + KNO_3 \rightarrow [NO_2NCSCo(C_2H_8N_2)_2]NO_3 + KCl$$

A. Werner and N. Gosling, Ann., **386**, 245 (1912)

20

$[Co(NCS)_2en_2]Cl$

KOH **III-469**

Trans diisothiocyanatodiethylenediamino cobaltic chloride is finely powdered and added to a small volume of potassium hydroxide solution (1:4) and triturated until a thin suspension is formed. The suspension is warmed until a violet colored solution results. This solution is cooled and crystals of trans hydroxoisothiocyanatodiethylenediamino cobaltic thiocyanate form.

$$[(NCS)_2Co(C_2H_8N_2)_2]Cl + KOH \rightarrow [OHNCSCo(C_2H_8N_2)_2]SCN + KCl$$

A. Werner and R. Schmidt, Ann., **386**, 160 (1912)

20

$[CoClNO_2en_2]Cl$

KSCN **III-470**

1 Gram of cis chloronitritodiethylenediamino cobaltic chloride is dissolved in 20 cc of water and warmed on the water bath. 3 grams of solid potassium thiocyanate is added and heating continued for 15 minutes. Upon cooling, red brown needles of cis nitritoisothio cyanatodiethylenediamino cobaltic thiocyanate deposit.

$$[ClNO_2Co(C_2H_8N_2)_2]Cl + 2KSCN \rightarrow [NO_2NCSCo(C_2H_8N_2)_2]SCN + 2KCl$$

A. Werner and C. Rix, Ann., **386**, 238 (1912)

20

$[Co(NH_3)_2Cl_2en]Cl$

KSCN **III-471**

Solid potassium thiocyanate is added to a concentrated aqueous solution of trans dichloroethylenediaminodiammino cobaltic chloride and trans dichloroethylenediaminodiammino cobaltic thiocyanate is precipitated as a bright green crystalline mass. It is dried on a porous plate.

$$[Cl_2Co(C_2H_8N_2)(NH_3)_2]Cl + KSCN \rightarrow [Cl_2Co(C_2H_8N_2)(NH_3)_2]SCN + KCl$$

A. Werner and F. Chaussy, Ann., **386**, 260 (1912) *20*

$[Co(NCS)_2en_2]Cl$

KSCN **III-472**

2 Grams of cis diisothiocyanatodiethylenediamino cobaltic chloride are dissolved in water acidified with acetic acid and solid potassium thiocyanate added. A precipitate of small red needles forms. This is dissolved in dilute acetic acid solution and allowed to crystallize. Cis diisothiocyanatodiethylenediamino cobaltic thiocyanate is obtained as lustrous red needles.

$$[(NCS)_2Co(C_2H_8N_2)_2]Cl + KSCN \rightarrow [(NCS)_2Co(C_2H_8N_2)_2]SCN + KCl$$

A. Werner and C. Rix, Ann., **386**, 198 (1912) *20*

$[CoNO_2NCSen_2]Cl$

KSCN **III-473**

3 Grams of cis nitritoisothiocyanatodiethylenediamino cobaltic chloride are dissolved in water containing acetic acid and solid potassium thiocyanate added. A brown crystalline precipitate forms. This is recrystallized from hot water and cis nitritoisothiocyanatodiethylenediamino cobaltic thiocyanate is obtained as brown plates.

$$[NO_2NCSCo(C_2H_8N_2)_2]Cl + KSCN \rightarrow [NO_2NCSCo(C_2H_8N_2)_2]SCN + KCl$$

A. Werner and C. Rix, Ann., **386**, 237 (1912) *20*

$[CoSO_3en_2]Cl$

KSCN **III-474**

5 Grams of sulfitodiethylenediamino cobaltic chloride are dissolved in 50 cc of water and the calculated amount of potassium thiocyanate added. The solution is concentrated on the water bath until crystallization begins and, as the solution cools, large brownish-red needles of sulfitodiethylenediamino cobaltic thiocyanate with two H_2O deposit. The compound is easily soluble in water.

$$[SO_3Co(C_2H_8N_2)_2]Cl + KSCN \rightarrow [SO_3Co(C_2H_8N_2)_2]SCN + (KCl)$$

A. Werner and M. Pokrowska, Ann., **386**, 84 (1912) *20*

$[CoCl_2en_2]Cl$

KSCN

$HClO_4$ **III-475**

HCl

A solution of trans dichlorodiethylenediamino cobaltic chloride is treated with potassium thiocyanate and an impure precipitate of cis chloroisothiocyanatodiethylenediamino cobaltic chloride forms. This bluish red precipitate is triturated with perchloric acid which dissolves it to form a dark red solution which soon sets to a red crystalline mass of the perchlorate. Alcohol is added and the residue removed by filtration, washed with alcohol and ether, then dissolved in a small volume of water and cooled. Hydrochloric acid is added and bluish red needles of cis chloroisothiocyanatodiethylenediamino cobaltic chloride deposit.

(a) $[Cl_2Co(C_2H_8N_2)_2]Cl + KSCN$
$\rightarrow [ClNCSCo(C_2H_8N_2)_2]Cl + KCl$

(b) $[ClNCSCo(C_2H_8N_2)_2]Cl + HClO_4$
$\rightarrow [ClNCSCo(C_2H_8N_2)_2]ClO_4 + HCl$

(c) $[ClNCSCo(C_2H_8N_2)_2]ClO_4 + HCl$
$\rightarrow [ClNCSCo(C_2H_8N_2)_2]Cl + HClO_4$

A. Werner and R. Schmidt, Ann., **386**, 140 (1912) *20*

$[Co(NH_3)_2Cl_2en]Cl$

K_2CO_3 **III-476**

1 Gram of trans dichloroethylenediaminodiammino cobaltic chloride and 1.06 grams of potassium carbonate are added to 8 cc of water and stirred until a clear solution results. This is heated over a free flame until the color changes to red and crystallization begins. As the solution cools, carbonatoethylenediaminodiammino cobaltic chloride deposits as a red crystalline powder.

$$[Cl_2Co(C_2H_8N_2)(NH_3)_2]Cl + K_2CO_3 \rightarrow [CO_3Co(C_2H_8N_2)(NH_3)_2]Cl + 2KCl$$

A. Werner and F. Chaussy, Ann., **386**, 261 (1912) *20*

$[CoSO_3en_2]Cl$

K_2PtCl_6 **III-477**

A concentrated aqueous solution of sulfitodiethylenediamino cobaltic chloride is treated with a 20% aqueous solution of potassium chloroplatinate and brown crystals of sulfitodiethylenediamino cobaltic chloroplatinate with four H_2O deposit.

$$2[SO_3Co(C_2H_8N_2)_2]Cl + K_2PtCl_6 + 4H_2O \rightarrow [SO_3Co(C_2H_8N_2)_2]_2PtCl_6 \cdot 4H_2O + (2KCl)$$

A. Werner and M. Pokrowska, Ann., **386**, 85 (1912) *20*

$[CoCl_2en_2]Cl$

LiCl **III-478**

5 Grams of trans dichlorodiethylenediamino cobaltic chloride are dissolved in 15 cc of water, filtered and 20 grams of solid lithium chloride added. A bright green fine crystalline powder precipitates. This is a double compound.

$$[Cl_2Co(C_2H_8N_2)_2]Cl + LiCl \rightarrow [Cl_2Co(C_2H_8N_2)_2]Cl \cdot LiCl$$

A. Werner and R. Bosshard, Ann., **386**, 105 (1912) *20*

$[CoCl_2en_2]Cl$

NH_3 **III-479**

Trans dichlorodiethylenediamino cobaltic chloride is treated with an absolute methyl alcoholic solution of ammonia and trans chloroammonodiethylenediamino cobaltic chloride is formed.

$$[Cl_2Co(C_2H_8N_2)_2]Cl + NH_3 \rightarrow [ClNH_3Co(C_2H_8N_2)_2]Cl_2$$

A. Werner and R. Schmidt, Ann., **386**, 170 (1912) *20*

$[CoCl_2en_2]Cl$

NH_3 **III-480**

NaBr

5 Grams of trans dichlorodiethylenediaminocobaltic chloride are added to 10 cc of liquid ammonia and the ammonia allowed to evaporate. A dark red residue remains that dissolves in water to form an orange red solution. This solution is filtered and a saturated aqueous solution of sodium bromide is added. After a short time yellow needles of a mixture of trans diamminodiethylenediaminocobaltic bromide and cis chloroamminodiethylenediaminocobaltic bromide separate.

$$2[Cl_2Co(C_2H_8N_2)_2]Cl + 3NH_3 + 5NaBr$$
$$\rightarrow [(NH_3)_2Co(C_2H_8N_2)_2]Br_3 + [NH_3ClCo(C_2H_8N_2)_2]Br_2 + 5NaCl$$

A. Werner and C. Rix, Ann., **386**, 201 (1912) *20*

$[CoNH_3Cl_2H_2Oen]Cl$

NH_3 **III-481**

1 Gram of trans dichloroaquoethylenediaminoamminocobaltic chloride is added to 3.5 cc of 25% ammonium hydroxide and triturated until solution is complete. After 5–10 minutes, 3.5 cc of concentrated hydrochloric acid is added and the solution heated until it becomes a greenish blue color. Green crystals are deposited as the solution cools. These are dissolved in a small volume of water and solid ammonium chloride added or, better, cool solution to minus 5°C and add hydrochloric acid. Green crystals of trans dichloroethylenediaminodiammino cobaltic chloride with one half H_2O deposit.

$$[Cl_2H_2OCo(C_2H_8N_2)NH_3]Cl + NH_3$$
$$\rightarrow [Cl_2Co(C_2H_8N_2)(NH_3)_2]Cl + H_2O$$

A. Werner and F. Chaussy, Ann., **386**, 257 (1912) *20*

$[CoC_2O_4en_2]Cl$

NH_3

KI

III-482

From the reaction mixture obtained when oxalatodiethylenediamino cobaltic chloride is reacted with ammonia under pressure and subsequently treated with potassium iodide, a brown crystalline deposit forms. The oxalate in this compound is not bound in the complex as it is precipitated by the addition of calcium chloride. Analysis of the compound proved it to be diamminodiethylenediamine cobaltic iodooxalate.

$$2[C_2O_4Co(C_2H_8N_2)_2]Cl + 2NH_3 + 4KI \rightarrow [Co(NH_3)_2(C_2H_8N_2)_2]_2 \cdot I_4C_2O_4 + (2KCl + K_2C_2O_4)$$

A. Werner and J. Rapiport, Ann., **386**, 78 (1912) *20*

$[CoClNO_2en_2]Cl$

NH_3

$Na_2S_2O_6$

III-483

Trans chloronitritodiethylenediamino cobaltic chloride is covered with liquid ammonia. The chloride dissolves to form a reddish yellow colored solution which changes to a brown color. The ammonia is allowed to evaporate and the residue dissolved in water acidified with acetic acid. Solid sodium dithionate is added and a precipitate forms which is dissolved in hot water acidified with acetic acid. Long prisms of trans nitritoamminodiethylenediamino cobaltic dithionate deposit as the solution cools.

(a) $[ClNO_2Co(C_2H_8N_2)_2]Cl + NH_3 \rightarrow [NO_2NH_3Co(C_2H_8N_2)_2]Cl_2$

(b) $[NO_2NH_3Co(C_2H_8N_2)_2]Cl_2 + Na_2S_2O_6 \rightarrow [NO_2NH_3Co(C_2H_8N_2)_2]S_2O_6 + 2NaCl$

A. Werner and C. Rix, Ann., **386**, 227 (1912) *20*

$[CoCl_2en_2]Cl$

NH_3 **III-484**

$Na_2S_2O_6$

3 Grams of acid free trans dichlorodiethylenediamino cobaltic chloride are added to a saturated methyl alcohol solution of ammonia and the solution shaken from time to time. The alcoholic solution becomes a deep red color. After twelve hours the residue is removed by suction, washed with ether, dried and dissolved in very dilute acetic acid. Solid sodium dithionate is added and trans chloroamminodiethylenediamino cobaltic dithionate deposits.

(a) $[Cl_2Co(C_2H_8N_2)_2]Cl + NH_3 \rightarrow [ClNH_3Co(C_2H_8N_2)_2]Cl_2$

(b) $[ClNH_3Co(C_2H_8N_2)_2]Cl_2 + Na_2S_2O_6$
$\rightarrow \underline{[ClNH_3Co(C_2H_8N_2)_2]S_2O_6} + 2NaCl$

A. Werner and R. Schmidt, Ann., **386**, 173 (1912)

20

$[CoCl_2en_2]Cl$

NH_4OH

NaBr **III-485**

2 Grams of cis dichlorodiethylenediamino cobaltic chloride (violeochloride) are dissolved in 10 cc concentrated ammonium hydroxide with the aid of gentle warming and the solution saturated with solid sodium bromide. A red precipitate forms which is recrystallized from aqueous solution by the addition of sodium bromide. Trans hydroxoaquodiethylenediamino cobaltic bromide is obtained as bright red crystals. The compound dissolves in water to form an intense red solution, but it is insoluble in alcohol.

$[Cl_2Co(C_2H_8N_2)_2]Cl + NH_4OH + 2NaBr + H_2O$
$\rightarrow [(H_2O)OHCo(C_2H_8N_2)_2]Br_2 + 2NaCl + NH_4Cl$

A. Werner and K. Lange, Ann., **386**, 97 (1912)

20

$[CoCl_2en_2]Cl$

$(NH_4)_2SO_4$ **III-486**

A concentrated aqueous solution of cis dichlorodiethylenediamino cobaltic chloride is saturated with ammonium sulfate and cis dichlorodiethylenediamino cobaltic sulfate precipitates.

$$2[Cl_2Co(C_2H_8N_2)_2]Cl + (NH_4)_2SO_4 \rightarrow [Cl_2Co(C_2H_8N_2)_2]_2SO_4 + 2NH_4Cl$$

A. Werner and R. Bosshard, Ann., **386**, 110 (1912) *20*

$[CoNO_2NCSen_2]Cl$

$(NH_4)_2SO_4$ **III-487**

Cis nitritoisothiocyanatodiethylenediamino cobaltic chloride is dissolved in water acidified with acetic acid and ammonium sulfate is added. A voluminous precipitate of cis nitritoisothiocyanatodiethylenediamino cobaltic sulfate forms. This precipitate is recrystallized from hot water containing acetic acid and the salt obtained as yellow lustrous crystals.

$$2[NO_2NCSCo(C_2H_8N_2)_2]Cl + (NH_4)_2SO_4 \rightarrow [NO_2NCSCo(C_2H_8N_2)_2]_2SO_4 + 2NH_4Cl$$

A. Werner and C. Rix, Ann., **386**, 235 (1912) *20*

$[CoClNCSen_2]Cl$

$NaNO_2$ **III-488**

A concentrated aqueous solution of one mole of cis chloroisothiocyanatodiethylenediamino cobaltic chloride is treated with 1 mole of sodium nitrite and the solution heated until it becomes yellow. The solution is cooled and needles of cis chloroisothiocyanatodiethylenediamino cobaltic nitrite are obtained in good yield.

$$[ClNCSCo(C_2H_8N_2)_2]Cl + NaNO_2 \rightarrow [ClNCSCo(C_2H_8N_2)_2]NO_2 + NaCl$$

A. Werner and C. Rix, Ann., **386**, 236 (1912) *20*

$[CoCl_2en_2]Cl$

$NaNO_2$ **III-489**

3 Grams of trans dichlorodiethylenediamino cobaltic chloride are dissolved in a small volume of water, filtered, acidified with a few drops of acetic acid and treated with an equimolecular weight of sodium nitrite, either in solid form or in concentrated solution. A green crystalline precipitate forms which is removed by filtration, washed with absolute alcohol then recrystallized from a small volume of water acidified with acetic acid by the addition of sodium nitrite. Trans dichlorodiethylenediamino cobaltic nitrite is obtained as small green crystals.

$$[Cl_2Co(C_2H_8N_2)_2]Cl + NaNO_2 \rightarrow [Cl_2Co(C_2H_8N_2)_2]NO_2 + NaCl$$

A. Werner and R. Bosshard, Ann., **386**, 106 (1912) *20*

$[CoCl_2en_2]Cl$

$NaNO_2$

HCl **III-490**

1 Mole of acid free trans dichlorodiethylenediamino cobaltic chloride is dissolved in the smallest possible quantity of cold water and a concentrated solution of one mole of sodium nitrite added, the solution stirred and allowed to stand. The solution soon sets to a semi-solid mass. This precipitate is filtered off, dissolved in the minimum quantity of water and concentrated hydrochloric acid added to the cooled solution. Pure cis dichlorodiethylenediamino cobaltic chloride deposits. 15 grams of the pure cis isomer was obtained from 70 grams of the trans salt.

(a) $[Cl_2Co(C_2H_8N_2)_2]Cl + NaNO_2$
$\rightarrow [Cl_2Co(C_2H_8N_2)_2]NO_2 + NaCl$

(b) $[Cl_2Co(C_2H_8N_2)_2]NO_2 + HCl \rightarrow [Cl_2Co(C_2H_8N_2)_2]Cl + HNO_2$

A. Werner and L. Cohn, Ann., **386**, 252 (1912) *20*

$[CoNO_2NCSen_2]Cl$

$NaNO_2$ **III-491**

3 Grams of trans nitritoisothiocyanatodiethylenediamino cobaltic chloride are dissolved in 24 cc of water and solid sodium nitrite added. A brown precipitate forms which is recrystallized from hot water and trans nitritoisothiocyanatodiethylenediamino cobaltic nitrite is deposited as large brown tablets with one H_2O.

$$[NO_2NCSCo(C_2H_8N_2)_2]Cl + NaNO_2 \rightarrow [NO_2NCSCo(C_2H_8N_2)_2]NO_2 + NaCl$$

A. Werner and N. Gosling, Ann., **386**, 246 (1912) *20*

$[Co(NH_3)_3Cl_2H_2O]Cl$

NaOH **III-492**

Trihydroxohexamminodicobaltic chloride is formed by action of sodium hydroxide solution on dichloroaquotriamminocobaltic monochloride.

$$2[Co(NH_3)_3(H_2O)Cl_2]Cl + 3NaOH \rightarrow [Co_2(OH)_3(NH_3)_6]Cl_3 + 3NaCl + 2H_2O$$

A. Werner, Ber., **40**, 4834 (1907)
Ref., J. J. Sudborough, J. Chem. Soc. (London), **94:2**, 43 (1908) *57*

$[CoCl_2en_2]Cl$

Na_2CO_3 **III-493**

5 Grams of trans dichlorodiethylenediamino cobaltic chloride are dissolved in 80 cc of water and added to a solution of 4.6 grams of sodium carbonate in 10 cc of water, heated on the steam bath for one hour while stirring vigorously and the solution filtered. During the heating the color of the solution changes from green to red. The solution is allowed to cool and the precipitate crystallized from hot water. Dark red, grooved, flat prismatic crystals of carbonato diethylenediamino cobaltic chloride with one H_2O deposit.

$$[Cl_2Co(C_2H_8N_2)_2]Cl + Na_2CO_3 \rightarrow [CO_3Co(C_2H_8N_2)_2]Cl + 2NaCl$$

A. Werner and J. Rapiport, Ann., **386**, 72 (1912) *20*
41

$[CoCl_2pn_2]Cl$

Na_2CO_3 **III-494**

3.8 Grams of trans dichloroditrimethylenediamino cobaltic chloride are dissolved in 10 cc of water and the calculated amount of sodium carbonate added. The solution is heated until it becomes bluish red. Alcohol is added and a white precipitate forms which is removed, and soon red needle like crystals of carbonatoditrimethylenediamino cobaltic chloride deposit. They may be recrystallized from water.

$$[Cl_2Co(C_3H_{10}N_2)_2]Cl + Na_2CO_3 \rightarrow [CO_3Co(C_3H_{10}N_2)_2]Cl + 2NaCl$$

A. Werner and G. Lindenberg, Ann., **386**, 271 (1912) *20*

$[CoCl_2en_2]Cl$

Na_2SO_3 **III-495**

10 Grams of cis dichlorodiethylenediamino cobaltic chloride are dissolved in 50 cc of water and treated with 10 grams of neutral sodium sulfite. The solution is heated to boiling and upon cooling, crystals of sulfitodiethylenediamino cobaltic chloride with one half H_2O deposit.

$$[Cl_2Co(C_2H_8N_2)_2]Cl + Na_2SO_3 \rightarrow [SO_3Co(C_2H_8N_2)_2]Cl + 2NaCl$$

A. Werner and M. Pokrowska, Ann., **386**, 81 (1912) *20*

$[CoCO_3en_2]Cl$

$Na_2S_2O_6$ **III-496**

A dilute solution of carbonatodiethylenediaminocobaltic chloride is treated with solid sodium dithionate. Dark red prisms of the corresponding complex dithionate are deposited.

$$2[CO_3Co(C_2H_8N_2)_2]Cl + Na_2S_2O_6 \rightarrow [CO_3Co(C_2H_8N_2)_2]_2S_2O_6 + 2NaCl$$

A. Werner, Ann., **386**, 76 (1912) *41*

20

$[Co(NH_3)_2Cl_2en]Cl$

$Na_2S_2O_6$

III-497

Solid sodium dithionate is added to a cold concentrated aqueous solution of trans dichloroethylenediaminodiammino cobaltic chloride and, in a short time, a bright green precipitate of trans dichloroethylenediamino diammino cobaltic dithionate forms. It is dried on a porous plate.

$$2[Cl_2Co(C_2H_8N_2)(NH_3)_2]Cl + Na_2S_2O_6 \rightarrow [Cl_2Co(C_2H_8N_2)(NH_3)_2]_2S_2O_6 + 2NaCl$$

A. Werner and F. Chaussy, Ann., **386**, 261 (1912)

20

$[Co(NH_3)_4(NO_2)_2]Cl$

$Na_3Co(NO_2)_6$

III-498

A solution of croceo cobalt chloride will react with a solution of sodium cobaltinitrite yielding orange red crystals of croceo cobaltocobaltic nitrite.

$$3[(NO_2)_2 \cdot Co(NH_3)_4]Cl + Na_3Co(NO_2)_6 \rightarrow [(NO_2)_2 \cdot Co(NH_3)_4]_3 \cdot (NO_2)_6 \cdot Co + 3NaCl$$

S. M. Jörgensen, Z. anorg. Chem., **5**, 147, (1894)

28

$[CoCl_2en_2]Cl$

$S(CH_2COOH)_2$

III-499

When a cold concentrated solution of dichlorodiethylenediamino cobaltic chloride is mixed with equimolar proportions of thiodiacetic acid, an addition compound is precipitated.

$$[Cl_2Co(NH_2C_2H_4NH_2)_2]Cl + 2S(CH_2COOH)_2 \rightarrow \underline{[Cl_2Co(NH_2C_2H_4NH_2)_2] \cdot OCOCH_2SCH_2COOH \cdot S(CH_2COOH)_2} + (HCl)$$

T. S. Price and S. A. Brazier, J. Chem. Soc. (London), **107**, 1735 (1915)

67

$[CoCl_2en_2]Cl$

Bromomalonic acid **III-500**

When a cold concentrated solution of dichlorodiethylenediamino cobaltic chloride is mixed with equimolar proportions of bromomalonic acid, an addition compound is precipitated.

$$[Cl_2Co(NH_2C_2H_4NH_2)_2]Cl + 2CHBr(COOH)_2 \rightarrow \underline{[Cl_2Co(NH_2C_2H_4NH_2)_2]\cdot OCOCHBrCOOH\cdot CHBr(COOH)_2} + (HCl)$$

T. S. Price and S. A. Brazier, J. Chem. Soc. (London), **107**, 1732 (1915) *57*

$[CoCl_2en_2]Cl$

Chloromalonic acid **III-501**

When a cold concentrated solution of dichlorodiethylenediamine cobaltic chloride is mixed with equimolar proportions of chloromalonic acid, an addition compound is precipitated.

$$[Cl_2Co(NH_2C_2H_4NH_2)_2]Cl + 2CHCl(COOH)_2 \rightarrow \underline{[Cl_2Co(NH_2C_2H_4NH_2)_2]\cdot OCOCHClCOOH\cdot CHCl(COOH)_2} + (HCl)$$

T. S. Price and S. A. Brazier, J. Chem. Soc. (London), **107**, 1733 (1915) *57*

$[CoCl_2en_2]Cl$

Dibromosuccinic acid **III-502**

When a cold concentrated solution of dichlorodiethylenediamine cobaltic chloride is mixed with equimolar proportions of dibromosuccinic acid, an addition compound is precipitated.

$$[Cl_2Co(NH_2C_2H_4NH_2)_2]Cl + (CHBr)_2(COOH)_2 \rightarrow \underline{[Cl_2Co(NH_2C_2H_4NH_2)_2]\cdot OCO(CHBr)_2COOH} + (HCl)$$

T. S. Price and S. A. Brazier, J. Chem. Soc. (London), **107**, 1734 (1915) *57*

$[CoCl_2en_2]Cl$

Dithioacetic acid **III-503**

When a cold concentrated solution of dichlorodiethylenediamine cobaltic chloride is mixed with equimolar proportions of dithiodiacetic acid, an addition compound is precipitated.

$$[Cl_2Co(NH_2C_2H_4NH_2)_2]Cl + S_2(CH_2COOH)_2 + H_2O \rightarrow \underline{[Cl_2Co(NH_2C_2H_4NH_2)_2]\cdot OCOCH_2S_2CH_2COOH\cdot H_2O} + (HCl)$$

T. S. Price and S. A. Brazier, J. Chem. Soc. (London), **107**, 1738 (1915) *57*

$[CoCl_2en_2]Cl$

Fumaric acid **III-504**

When a cold concentrated solution of dichlorodiethylenediamino cobaltic chloride is mixed with equimolar proportions of fumaric acid, an addition compound is precipitated.

$$[Cl_2Co(NH_2C_2H_4NH_2)_2]Cl + C_2H_2(COOH)_2 \rightarrow \underline{[Cl_2Co(NH_2C_2H_4NH_2)_2]\cdot OCOC_2H_2COOH} + (HCl)$$

T. S. Price and S. A. Brazier, J. Chem. Soc. (London), **107**, 1739 (1915) *57*

$[CoCl_2en_2]Cl$

Succinic acid **III-505**

When a cold concentrated solution of dichlorodiethylenediamine cobaltic chloride is mixed with equimolar proportions of succinic acid, an addition compound is precipitated.

$$[Cl_2Co(NH_2C_2H_4NH_2)_2]Cl + C_2H_4(COOH)_2 \rightarrow \underline{[Cl_2Co(NH_2C_2H_4NH_2)_2]\cdot OCOC_2H_4COOH} + (HCl)$$

T. S. Price and S. A. Brazier, J. Chem. Soc. (London), **107**, 1734 (1915) *57*

$[Co(NH_3)_3C_2O_4H_2O]Cl$

Decomp. **III-506**

The violet red color of oxalatoaquotriamminocobaltic chloride changes to an intense violet after standing some time and the solubility in water decreases. The aquo salt changes to cobaltic chlorooxalatotriammine.

$$[(C_2O_4)H_2OCo(NH_3)_3]Cl \rightarrow [(C_2O_4)CoCl(NH_3)_3] + H_2O$$

A. Werner, E. Bindschedler and C. Sakur, Ann., **405**, 231 (1914) *20*

$[CoCl_2en_2]Cl$

Δ

KSCN **III-507**

20 Grams of acid free trans dichlorodiethylenediamino cobaltic chloride are heated to 100°C and dissolved in 30 cc of water with the aid of heat. A solution of 6.8 grams of potassium thiocyanate is added and a bright green crystalline mass precipitates. The solution is heated slowly until it just begins to boil then cooled and allowed to stand two hours. A large amount of bluish red crystals of cis chloroisothiocyanatodiethylenediamino cobaltic chloride deposit. These are removed by suction filtration and 6.8 grams of solid potassium thiocyanate added to the filtrate. The solution is stirred until all of the salt dissolves and dark violet crystals of cis chloroisothiocyanatodiethylenediamino cobaltic thiocyanate deposit. These may be recrystallized from hot water.

(a) $[Cl_2Co(C_2H_8N_2)_2]Cl + KSCN$
$$\rightarrow [ClNCSCo(C_2H_8N_2)_2]Cl + KCl$$

(b) $[Cl_2Co(C_2H_8N_2)_2]Cl + 2KSCN$
$$\rightarrow [ClNCSCo(C_2H_8N_2)_2]SCN + 2KCl$$

A. Werner and R. Schmidt, Ann., **386**, 133 (1912) *20*

$[CoCl_2en_2]Cl$

Δ **III-508**

KSCN

$HClO_4$

An acid free solution of trans dichlorodiethylenediamino cobaltic chloride is treated with solid potassium thiocyanate and bright green trans dichlorodiethylenediamino cobaltic thiocyanate precipitates. This salt is removed by filtration and washed with water. It is then made into a thick paste with water and heated over a free flame until the color becomes darker and finally deep violet. The solution is filtered, cooled and perchloric acid added. Trans chloroisothiocyanatodiethylenediamino cobaltic perchlorate is precipitated.

(a) $[Cl_2Co(C_2H_8N_2)_2]Cl + KSCN \rightarrow [Cl_2Co(C_2H_8N_2)_2]SCN + KCl$

(b) $[Cl_2Co(C_2H_8N_2)_2]SCN + HClO_4$
$\rightarrow [ClNCSCo(C_2H_8N_2)_2]ClO_4 + HCl$

A. Werner and R. Schmidt, Ann., **386**, 136 (1912) *20*

$[CoCl_2en_2]Cl$

Δ **III-509**

$(NH_4)_2SO_4$

10 Grams of neutral trans dichlorodiethylenediamino cobaltic chloride are added to 20 cc of water and the mixture heated until solution is complete and the color is dark blue. This solution is allowed to stand for one hour then 10 grams of solid ammonium sulfate is added and stirred until dissolved. This solution is allowed to stand 12 hours when a bluish red crystalline crust forms. This crystalline mass is separated and extracted with 10 cc portions of cold water when the green salt goes into solution. The residue is cis chloroaquodiethylenediamino cobaltic sulfate and it is washed with cold water until the washings are a pure red color. The salt crystallizes with one and one half H_2O.

(a) $[Cl_2Co(C_2H_8N_2)_2]Cl + H_2O \rightarrow [Cl(H_2O)Co(C_2H_8N_2)_2]Cl_2$

(b) $[Cl(H_2O)Co(C_2H_8N_2)_2]Cl_2 + (NH_4)_2SO_4$
$\rightarrow [Cl(H_2O)Co(C_2H_8N_2)_2]\cdot SO_4 + 2NH_4Cl$

A. Werner, L. Gerb, S. Lorie and J. Rapiport, Ann., **386**, 122 (1912) *20*

$[CoNH_3Clen_2]Cl \cdot ClO_4$

NH_3 **III-510**

Trans chloroamminodiethylenediamino cobaltic chloride perchlorate is dissolved in liquid ammonia. The compound dissolves to form a blue red solution but changes slowly to yellowish red. The ammonia is allowed to evaporate and the residue consists of a mixture of trans and cis diammonodiethylenediamino cobaltic chloride perchlorate.

$$[ClNH_3Co(C_2H_8N_2)_2]Cl \cdot ClO_4 + NH_3 \rightarrow [(NH_3)_2Co(C_2H_8N_2)_2]Cl_2 \cdot ClO_4$$

A. Werner and R. Schmidt, Ann., **386**, 174 (1912) *20*

$[CoNH_3Clen_2]Cl \cdot ClO_4$

$NaNO_2$ **III-511**

Solid sodium nitrite is added to a solution of trans chloroamminodiethylenediamino cobaltic chloride perchlorate which has been acidified with acetic acid and trans chloroamminodiethylenediamino cobaltic nitrite deposits as a red crystalline powder.

$$[ClNH_3Co(C_2H_8N_2)_2]Cl \cdot ClO_4 + 2NaNO_2 \rightarrow [ClNH_3Co(C_2H_8N_2)_2](NO_2)_2 + NaCl + NaClO_4$$

A. Werner and R. Schmidt, Ann., **386**, 173 (1912) *20*

$[CoNH_3Clen_2]Cl \cdot ClO_4$

$NaNO_2$ **III-512**

Solid sodium nitrite is added to a neutral aqueous solution of trans chloroamminodiethylenediamino cobaltic chloride perchlorate. Ammonia is evolved and the solution soon sets to a crystalline mass of trans chloronitritodiethylenediamino cobaltic nitrite.

$$[ClNH_3Co(C_2H_8N_2)_2]Cl \cdot ClO_4 + 2NaNO_2 \rightarrow [ClNO_2Co(C_2H_8N_2)_2]NO_2 + NH_3 + NaCl + NaClO_4$$

A. Werner and R. Schmidt, Ann., **386**, 174 (1912) *20*

$[CoClNCSen_2]ClO_4$

H_2O_2

HCl

III-513

5 Grams of trans chloroisothiocyanatodiethylenediamino cobaltic perchlorate is shaken with 10 cc of water, 3 cc of dilute sulphuric acid (1:3) and 20 grams of concentrated hydrogen peroxide. The perchlorate dissolves slowly so the mixture is warmed until solution is complete, then cooled. This solution is allowed to stand for 1 hour then concentrated hydrochloric acid added and concentrated on the water bath. Some green dichlorodiethylenediamino cobaltic chloride separates. This is filtered off and the filtrate cooled. Bright red lustrous leaflets of trans chloroamminodiethylenediamino cobaltic chloride perchlorate are deposited.

$$[ClNCSCo(C_2H_8N_2)_2]ClO_4 + 4H_2O_2 + HCl \rightarrow [ClNH_3Co(C_2H_8N_2)_2]Cl \cdot ClO_4 + H_2SO_4 + CO_2 + 2H_2O$$

A. Werner and R. Schmidt, Ann., **386**, 171 (1912) *20*

$[CoClNCSen_2]ClO_4$

$LiNO_3$

III-514

A concentrated aqueous solution of cis chloroisothiocyanatodiethylenediamino cobaltic perchlorate is treated with solid lithium nitrate and the solution stirred until all solid matter has dissolved. Cis chloroisothiocyanatodiethylenediamino cobaltic nitrate is precipitated as a dark red difficultly soluble powder. The salt is recrystallized from hot water and dark bluish red needles are obtained.

$$[ClNCSCo(C_2H_8N_2)_2] \cdot ClO_4 + LiNO_3 \rightarrow [ClNCSCo(C_2H_8N_2)_2] \cdot NO_3 + LiClO_4$$

A. Werner and R. Schmidt, Ann., **386**, 141 (1912) *20*

$[CoBrNCSen_2]ClO_4$

$LiNO_3$ **III-515**

The solution of cis bromoisothiocyanatodiethylenediamino cobaltic perchlorate, obtained by the action of potassium thiocyanate and perchloric acid on trans dibromodiethylenediamine cobaltic bromide, is warmed to 60–70°C, quickly filtered, lithium nitrate added, stirred to dissolve and cooled. The difficultly soluble violet brown cis bromoisothiocyanatodiethylenediamino cobaltic nitrate precipitates. The salt may be recrystallized from warm water if one works with small portions at a time because of the ease of formation of the cis aquoisothiocyanato salt.

$$[BrNCSCo(C_2H_8N_2)_2]ClO_4 + LiNO_3 \rightarrow [BrNCSCo(C_2H_8N_2)_2]NO_3 + LiClO_4$$

A. Werner and R. Schmidt, Ann., **386**, 151 (1912)

20

$[CoBrNCSen_2]ClO_4$

$(NH_4)_2SO_4$ **III-516**

The solution of cis bromoisothiocyanatodiethylenediamine cobaltic perchlorate prepared by treating trans dibromodiethylenediamine cobaltic bromide with potassium thiocyanate and perchloric acid is treated with ammonium sulfate and cis bromoisothiocyanato-diethylenediamine cobaltic sulfate deposits as reddish lilac, fine, leafy silky crystals which are only slightly soluble in water.

$$2[BrNCSCo(C_2H_8N_2)_2]ClO_4 + (NH_4)_2SO_4 \rightarrow [BrNCSCo(C_2H_8N_2)_2]_2SO_4 + 2NH_4ClO_4$$

A. Werner and R. Schmidt, Ann., **386**, 152 (1912)

20

$[CoClNCSen_2]ClO_4$

Na_2SO_4 **III-517**

Solid sodium sulfate is added to an aqueous solution of cis chloroisothiocyanatodiethylenediamino cobaltic perchlorate and cis chloroisothiocyanatodiethylenediamino cobaltic sulfate precipitates. It is recrystallized from warm water and obtained as a violet red powder.

$$2[ClNCSCo(C_2H_8N_2)_2]ClO_4 + Na_2SO_4 \rightarrow [ClNCSCo(C_2H_8N_2)_2]_2SO_4 + 2NaClO_4$$

A. Werner and R. Schmidt, Ann., **386**, 141 (1912) *20*

$[CoClNCSen_2]ClO_4$

$Na_2S_2O_6$ **III-518**

Solid sodium dithionate is added to a concentrated aqueous solution of cis chloroisothiocyanatodiethylenediamino cobaltic perchlorate and difficultly soluble cis chloroisothiocyanatodiethylenediamino cobaltic dithionate precipitates. The precipitate is recrystallized from hot water and the salt is obtained as brownish red crystals with one H_2O. During crystallization the solution must not be heated too hot nor too long as this favors the formation of a large amount of the aquoisothiocyanato salt.

$$2[ClNCSCo(C_2H_8N_2)_2]ClO_4 + Na_2S_2O_6 \rightarrow [ClNCSCo(C_2H_8N_2)_2]_2S_2O_6 + 2NaClO_4$$

A. Werner and R. Schmidt, Ann., **386**, 140 (1912) *20*

$[CoNH_3Clen_2](ClO_4)_2$

$Na_2S_2O_6$ **III-519**

Solid sodium dithionate is added to an aqueous solution of trans chloroamminodiethylenediamino cobaltic perchlorate and the solution stirred until all is in solution. Insoluble bright, red, fine needles of trans chloroamminodiethylenediamino cobaltic dithionate with one H_2O precipitate. These are recrystallized from hot water and the compound is obtained as long, bright, blue red, thin very characteristic needles.

$$[ClNH_3Co(C_2H_8N_2)_2](ClO_4)_2 + Na_2S_2O_6 \rightarrow [ClNH_3Co(C_2H_8N_2)_2]S_2O_6 + 2NaClO_4$$

A. Werner and R. Schmidt, Ann., **386**, 172 (1912) *20*

$[Co(NH_3)_5Cl]Cl_2$

$C_2H_4(NH_2)_2$ **III-520**

HBr

Three moles of ethylenediamine are added to one mole of chloropentamminocobalt chloride in a flask fitted with an air condenser. The mixture is warmed until it assumes a yellowish color. The yellow mass is dissolved in water, filtered and concentrated hydrobromic acid added to the filtrate. A thick precipitate forms which is removed by filtration, dissolved in water and again treated with concentrated hydrobromic acid. The residue is removed by filtration and dried. It is then crystallized from water and yellow needles of diethylenediammocobalt bromide separate. They contain three H_2O.

$$[(NH_3)_5CoCl]Cl_2 + 2C_2H_4(NH_2)_2 + 3HBr + 2H_2O \rightarrow Co[C_2H_4(NH_2)_2]_2Br_3 + 3NH_4Cl + 2NH_4OH$$

P. Pfeiffer, Ann., **346**, 59 (1906)

20

$C[o(NH_3)_5Cl]Cl_2$

$C_2H_4(NH_2)_2$ **III-521**

5 Grams of chloropentamminecobalt chloride are mixed with 5.4 g. of ethylenediamine in a flask fitted with an air condenser. The mixture is warmed and heating continued until the mass assumes a yellowish color. It is then dissolved in water and treated with a solution of potassium iodide. The yellow precipitate is recrystallized from water and yellow needles of triethylenediaminocobalt iodide are obtained.

$$[(NH_3)_5CoCl]Cl_2 + 3C_2H_4(NH_2)_2 + 3KI + 5H_2O \rightarrow [Co(C_2H_4(NH_2)_2)_3]I_3 + 3KCl + 5NH_4OH$$

P. Pfeiffer and T. Gassmann, Ann., **346**, 60 (1906)

20

$[Co(NH_3)_5Cl]Cl_2$

$C_2H_4(NH_2)_2$

KSCN **III-522**

5 Grams of chloropentaminecobalt chloride is mixed with 5.3 grams of ethylenediamine in a flask fitted with an air condenser. The flask and contents are heated on the water bath for 8 hours. The yellowish mass is dissolved in as little water as possible, filtered and the filtrate treated with a solution of potassium thiocyanate. The yellow precipitate is removed by filtration and crystallized from hot water. Dark yellow compact crystals of triethylenediaminocobalt thiocyanate are formed.

$$[(NH_3)_5CoCl]Cl_2 + 3C_2H_4(NH_2)_2 + 3KSCN + 5H_2O \rightarrow [Co(C_2H_4(NH_2)_2)_3]\cdot[(SCN)_3] + 3KCl + 5NH_4OH$$

P. Pfeiffer and T. Gassmann, Ann., **346**, 61 (1906) *20*

$[Co(NH_3)_5Cl]Cl_2$

$C_3H_6(NH_2)_2$ **III-523**

1 Mole of chloropentamminecobalt chloride is added to 3 moles of propylenediamine in a flask fitted with an air condenser and the solution heated on the water bath until a yellow color is assumed. Ammonia is evolved. The mass is dissolved in water and filtered. The filtrate is a solution of tripropylenediaminocobalt chloride.

$$[(NH_3)_5CoCl]Cl_2 + 3C_3H_6(NH_2)_2 \rightarrow [Co(C_3H_6(NH_2)_2)_3]Cl_3 + 5NH_3$$

P. Pfeiffer and T. Gassmann, Ann., **346**, 62 (1906) *20*

$[CoH_2OClen_2]Cl_2$

HBr **III-524**

50 Grams of chloroaquodiethylenediamino cobaltic chloride are triturated with 50 cc of concentrated hydrobromic acid and allowed to stand 20 minutes. The solution is reddish violet and a dark violet red precipitate forms. This is removed by filtration, washed with alcohol and ether, dissolved in 250 cc of cold water and 150 cc of concentrated hydrobromic acid added. Cis chloroaquodiethylenediamino cobaltic bromide deposits as small reddish violet leafy crystals with one H_2O. The salt is soluble in water.

$$[Cl(H_2O)Co(C_2H_8N_2)_2]Cl_2 + 2HBr \rightarrow [Cl(H_2O)Co(C_2H_8N_2)_2]Br_2 + 2HCl$$

A. Werner, L. Gerb, S. Lorie and J. Rapiport, Ann., **386**, 124 (1912) *20*

$[CoNH_3NO_2en_2]Cl_2$

HCl **III-525**

A solution of trans nitritoamminodiethylenediamino cobaltic chloride in concentrated hydrochloric acid is, when heated, converted to trans chloroammonodiethylenediamino cobaltic chloride.

$$[NO_2NH_3Co(C_2H_8N_2)_2]Cl_2 + HCl \rightarrow [ClNH_3Co(C_2H_8N_2)_2]Cl_2 + HNO_2$$

A. Werner and R. Schmidt, Ann., **386**, 169 (1912) *20*

$[ClCo(C_2H_8N_2)_2(C_3H_7N)]Cl_2$

HNO_3 **III-526**

When an aqueous solution of monochlorodiethylenediaminoallylamino cobaltic chloride is treated with concentrated nitric acid crystals of monochlorodiethylenediaminoallylamino cobaltic nitrate are formed.

$$[ClCo(C_2H_8N_2)_2(C_3H_7N)]Cl_2 + 2HNO_3 \rightarrow [ClCo(C_2H_8N_2)_2(C_3H_7N)](NO_3)_2 + 2HCl$$

J. Meisenheimer and E. Kinderlen, Ann., **438**, 250 (1924) *20*

$[CoNH_3Clen_2]Cl_2$

HNO_3 **III-527**

Nitric acid is added to a solution of trans chloroamminodiethylenediamino cobaltic chloride and alcohol added until a permanent turbidity results. Trans chloroamminodiethylenediamino cobaltic nitrate precipitates.

$$[ClNH_3Co(C_2H_8N_2)_2]Cl_2 + 2HNO_3 \rightarrow [ClNH_3Co(C_2H_8N_2)_2](NO_3)_2 + 2HCl$$

A. Werner and R. Schmidt, Ann., **386**, 173 (1912) *20*

$[CoH_2ONCSen_2]Cl_2$

H_2O_2

HCl

III-528

Trans aquoisothiocyanatodiethylenediamino cobaltic chloride and other salts of this series are oxidized by hydrogen peroxide in the presence of hydrochloric acid. The chloride of this series gives trans chloroamminodiethylenediamino cobaltic chloride.

$$[H_2ONCSCo(C_2H_8N_2)_2]Cl_2 + 4H_2O_2 + HCl \rightarrow [ClNH_3Co(C_2H_8N_2)_2]Cl_2 + CO_2 + 3H_2O + H_2SO_4$$

A. Werner and R. Schmidt, Ann., **386**, 162 (1912)

20

$[ClCo(C_2H_8N_2)_2(C_3H_7N)]Cl_2$

KBr

III-529

Monochlorodiethylenediaminoallylamino cobaltic chloride is suspended in water and an excess of solid potassium bromide added and the solution shaken. Dark colored plates of monochlorodiethylenediaminoallylamino cobaltic bromide are formed.

$$[ClCo(C_2H_8N_2)_2(C_3H_7N)]Cl_2 + 2KBr \rightarrow [ClCo(C_2H_8N_2)_2(C_3H_7N)]Br_2 + 2KCl$$

J. Meisenheimer and E. Kinderlen, Ann., **438**, 249 (1924)

20

$[ClCo(C_2H_8N_2)_2(C_7H_9N)]Cl_2$

KBr

III-530

Powdered potassium bromide is added to a concentrated aqueous solution of monochlorodiethylenediaminobenzylamino cobaltic chloride and monochlorodiethylenediaminobenzylamino cobaltic bromide precipitates as a lilac colored crystalline material which has one half H_2O. It may be recrystallized from aqueous solution by the addition of potassium bromide.

$$[ClCo(C_2H_8N_2)_2(C_7H_9N)]Cl_2 + 2KBr \rightarrow [ClCo(C_2H_8N_2)_2(C_7H_9N)]Br_2 + 2KCl$$

J. Meisenheimer and E. Kinderlen, Ann., **438**, 252 (1924)

20

$[Cl(C_2H_8N_2)_2Co(C_6H_5NH_2)]Cl_2$

KBr **III-531**

2 Grams of monochlorodiethylenediaminoanilino cobaltic chloride is dissolved in 70 cc of cold water and an excess (4 moles) of solid potassium bromide added. Monochlorodiethylenediaminoanilino cobaltic bromide deposits as reddish violet prisms with one half H_2O. The crystals are washed with 50% alcohol, 95% alcohol and then ether. Yield 2 grams. The compound may be recrystallized from aqueous solution by the addition of concentrated hydrobromic acid.

$$[Cl(C_2H_8N_2)_2Co(C_6H_5NH_2)]Cl_2 + 2KBr \rightarrow [Cl(C_2H_8N_2)_2Co(C_6H_5NH_2)]Br_2 + 2KCl$$

J. Meisenheimer and E. Kinderlen, Ann., **438**, 244 (1924) *20*

$[Cl(C_2H_8N_2)_2Co(C_6H_5NH_2)]Cl_2$

KI **III-532**

Monochlorodiethylenediaminoanilino cobaltic chloride is dissolved in water and solid potassium iodide added in considerable excess. Monochloroethylenediaminoanilino cobaltic iodide precipitates as thick red prisms which decompose at 40–5°C.

$$[Cl(C_2H_8N_2)_2Co(C_6H_5NH_2)]Cl_2 + 2KI \rightarrow [Cl(C_2H_8N_2)_2Co(C_6H_5NH_2)]I_2 + 2KCl$$

J. Meisenheimer and E. Kinderlen, Ann., **438**, 244 (1924) *20*

$[ClCo(C_2H_8N_2)_2(C_3H_7N)]Cl_2$

KI **III-533**

Monochlorodiethylenediaminoallylamino cobaltic chloride is suspended in water and solid potassium iodide added. The mixture is shaken and crystals of monochlorodiethylenediaminoallylamino cobaltic iodide are formed. The compound is deposited as dark violet plates. They are insoluble in aqueous solutions of potassium iodide.

$$[ClCo(C_2H_8N_2)_2(C_3H_7N)]Cl_2 + 2KI \rightarrow [ClCo(C_2H_8N_2)_2(C_3H_7N)]I_2 + 2KCl$$

J. Meisenheimer and E. Kinderlen, Ann., **438**, 249 (1924) *20*

$[ClCo(C_2H_8N_2)_2(C_7H_9N)]Cl_2$

KI **III-534**

Powdered potassium iodide is added to a concentrated aqueous solution of monochlorodiethylenediaminobenzylamino cobaltic chloride and monochlorodiethylenediaminobenzylamino cobaltic iodide is precipitated as brownish red six sided tablets which are moderately soluble in warm water but only very slightly soluble in aqueous solutions of potassium iodide.

$$[ClCo(C_2H_8N_2)_2(C_7H_9N)]Cl_2 + 2KI \rightarrow [ClCo(C_2H_8N_2)_2(C_7H_9N)]I_2 + 2KCl$$

J. Meisenheimer and E. Kinderlen, Ann., **438**, 252 (1924) *20*

$[(C_3H_6O_2N)Co(C_2H_8N_2)_2]Cl_2$

KI **III-535**

One gram of sarcosine (methylaminoacetic acid) diethylenediamino cobaltic chloride is dissolved in 3 cc of hot water and one gram of solid potassium iodide added. Red needles of sarcosine diethylenediamino cobaltic iodide deposit. Yield 1.2 grams. It may be recrystallized from 2 cc of hot water. The crystals have one H_2O.

$$[(C_3H_6O_2N)Co(C_2H_8N_2)_2]Cl_2 + 2KI \rightarrow [(C_3H_6O_2N)Co(C_2H_8N_2)_2]I_2 + 2KCl$$

J. Meisenheimer and E. Kinderlen, Ann., **438**, 270 (1924) *20*

$[CoNH_3Clen_2]Cl_2$

KSCN **III-536**

5 Grams of cis chloroamminodiethylenediamino cobaltic chloride are dissolved in water to form a concentrated solution and solid potassium thiocyanate added. The solution is warmed slightly and warming repeated during 2 hours when 3 grams of trans chloroisothiocyanatodiethylenediamino cobaltic thiocyanate has deposited. Ammonia is evolved during the reaction.

$$[ClNH_3Co(C_2H_8N_2)_2]Cl_2 + 2KSCN \rightarrow [ClNCSCo(C_2H_8N_2)_2]SCN + 2KCl + NH_3$$

A. Werner and R. Schmidt, Ann., **386**, 169 (1912) *20*

$[CoNH_3Clen_2]Cl_2$

KSCN **III-537**

Solid potassium thiocyanate is added to a concentrated aqueous solution of trans chloroamminodiethylenediamino cobaltic chloride and the solution warmed slightly. Ammonia is evolved and violet trans chloroisothiocyanatodiethylenediamino cobaltic thiocyanate precipitates.

$$[ClNH_3Co(C_2H_8N_2)_2]Cl_2 + 2KSCN \rightarrow \underline{[ClNCSCo(C_2H_8N_2)_2]SCN} + 2KCl + (NH_3)$$

A. Werner and R. Schmidt, Ann., **386**, 175 (1912) *20*

$[CoNH_3Clen_2]Cl_2$

NH_3 **III-538**

Cis chloroamminodiethylenediamino cobaltic chloride is added to liquid ammonia, allowed to stand two hours then the ammonia allowed to evaporate. Yellow diamminodiethylenediamino cobaltic chloride remains as a residue.

$$[ClNH_3Co(C_2H_8N_2)_2]Cl_2 + NH_3 \rightarrow [(NH_3)_2Co(C_2H_8N_2)_2]Cl_3$$

A. Werner and R. Schmidt, Ann., **386**, 167 (1912) *20*

$[CoNH_3Clen_2]Cl_2$

$NaClO_4$ **III-539**

Solid sodium perchlorate is added to a concentrated aqueous solution of cis chloroamminodiethylenediamino cobaltic chloride and red oblong prisms of cis chloroamminodiethylenediamino cobaltic chloride perchlorate deposit. These may be recrystallized from hot water.

$$[ClNH_3Co(C_2H_8N_2)_2]Cl_2 + NaClO_4 \rightarrow [ClNH_3Co(C_2H_8N_2)_2]Cl \cdot ClO_4 + NaCl$$

A. Werner and R. Schmidt, Ann., **386**, 166 (1912) *20*

$[CoH_2OClen_2]Cl_2$

$NaNO_2$ **III-540**

17 Grams of cis chloroaquodiethylenediamino cobaltic chloride are dissolved in 40 cc of cold water, the solution filtered and 7 grams of solid sodium nitrite added. A dark violet precipitate forms which is filtered off by suction, dissolved in 45 cc of cold water and 9 grams of solid sodium nitrite added. Monoclinic crystals of cis chloroaquodiethylenediamino cobaltic nitrite deposit. The yield is 10 grams and the salt is water soluble.

$$[Cl(H_2O)Co(C_2H_8N_2)_2]Cl_2 + 2NaNO_2 \rightarrow [Cl(H_2O)Co(C_2H_8N_2)_2](NO_2)_2 + 2NaCl$$

A. Werner, L. Gerb, S. Lorie and J. Rapiport, Ann., **386**, 125 (1912) *20*

$[CoNH_3Clen_2]Cl_2$

$NaNO_2$ **III-541**

20 Grams of cis chloroamminodiethylenediamino cobaltic chloride are dissolved in 70 cc of cold water, filtered acidified with acetic acid and 30 cc of a concentrated aqueous solution of sodium nitrite together with 20 grams of solid sodium nitrite are added. Cherry red crystals of cis chloroamminodiethylenediamino cobaltic nitrite deposit.

$$[ClNH_3Co(C_2H_8N_2)_2]Cl_2 + 2NaNO_2 \rightarrow [ClNH_3Co(C_2H_8N_2)_2](NO_2)_2 + 2NaCl$$

A. Werner and R. Schmidt, Ann., **386**, 167 (1912) *20*

$[Cl(C_2H_8N_2)_2Co(C_6H_5NH_2)]Cl_2$

$NaNO_3$ **III-542**

2 Grams of chlorodiethylenediaminoanilino cobaltic chloride are dissolved in 50 cc of water and 4–5 mole equivalents of pulverized sodium nitrate added while shaking. Micro crystals of chlorodiethylenediaminoanilino cobaltic nitrate are formed. After one half hour the crystals are filtered off and washed with dilute nitric acid. The compound is soluble in water and concentrated nitric acid.

$$[Cl(C_2H_8N_2)_2Co(C_6H_5NH_2)]Cl_2 + 2NaNO_3 \rightarrow [Cl(C_2H_8N_2)_2Co(C_6H_5NH_2)](NO_3)_2 + 2NaCl$$

J. Meisenheimer and E. Kinderlen, Ann., **438**, 245 (1924) *20*

$[ClCo(C_2H_8N_2)_2(C_7H_9N)]Cl_2$

$NaNO_3$ **III-543**

2 Grams of chlorodiethylenediamino-p-toluidino cobaltic chloride are dissolved in 40 cc of water by warming slightly. One gram of sodium nitrate is added and the solution allowed to stand overnight. Six sided violet red prisms of chlorodiethylenediamino-p-toluidino cobaltic chloronitrate are formed.

$$[ClCo(C_2H_8N_2)_2(C_7H_9N)]Cl_2 + NaNO_3 \rightarrow [ClCo(C_2H_8N_2)_2(C_7H_9N)]Cl \cdot NO_3 + NaCl$$

J. Meisenheimer and E. Kinderlen, Ann., **438**, 247 (1924) *20*

$[CoNH_3Clen_2]Cl_2$

$Na_2Cr_2O_7$ **III-544**

Solid potassium dichromate is added to an aqueous solution of trans chloroamminodiethylenediamino cobaltic chloride and the mixture rubbed. The residue is crystallized from water and trans chloroamminodiethylenediamino cobaltic dichromate is obtained as orange red crystals.

$$[ClNH_3Co(C_2H_8N_2)_2]Cl_2 + Na_2Cr_2O_7 \rightarrow [ClNH_3Co(C_2H_8N_2)_2]Cr_2O_7 + 2NaCl$$

A. Werner and R. Schmidt, Ann., **386**, 172 (1912) *20*

$[ClCo(C_2H_8N_2)_2(C_3H_7N)]Cl_2$

$Na_2S_2O_6$ **III-545**

Solid sodium dithionate is added to an aqueous suspension of monochlorodiethylenediaminoallylamino cobaltic chloride and the solution warmed slightly. Very small violet rose colored crystals of monochloroethylenediaminoallylaminocobaltic dithionate are deposited. The compound is almost insoluble in water.

$$[ClCo(C_2H_8N_2)_2(C_3H_7N)]Cl_2 + Na_2S_2O_6 \rightarrow [ClCo(C_2H_8N_2)_2(C_3H_7N)]S_2O_6 + 2NaCl$$

J. Meisenheimer and E. Kinderlen, Ann., **438**, 249 (1924) *20*

$[ClCo(C_2H_8N_2)_2(C_7H_9N)]Cl_2$

$Na_2S_2O_6$ **III-546**

Solid sodium dithionate is added in excess to an aqueous suspension of chlorodiethylenediamino-p-toluidino cobaltic chloride and chlorodiethylenediamino-p-toluidino cobaltic dithionate is deposited as very fine brownish rose needles.

$$[ClCo(C_2H_8N_2)_2(C_7H_9N)]Cl_2 + Na_2S_2O_6 \rightarrow [ClCo(C_2H_8N_2)_2(C_7H_9N)]S_2O_6 + 2NaCl$$

J. Meisenheimer and E. Kinderlen, Ann., **438**, 247 (1924) *20*

$[ClCo(C_2H_8N_2)_2(C_7H_9N)]Cl_2$

$Na_2S_2O_6$ **III-547**

Monochlorodiethylenediaminobenzylamino cobaltic chlorodithionate is precipitated when solid sodium dithionate is added to a concentrated aqueous solution of monochlorodiethylenediaminobenzylamino cobaltic chloride. The compound is obtained as raspberry red crystals.

$$2[ClCo(C_2H_8N_2)_2(C_7H_9N)]Cl_2 + Na_2S_2O_6 \rightarrow [ClCo(C_2H_8N_2)_2(C_7H_9N)]_2Cl_2S_2O_6 + 2NaCl$$

J. Meisenheimer and E. Kinderlen, Ann., **438**, 252 (1924) *20*

$[CoNH_3Clen_2]Cl_2$

$Na_2S_2O_6$ **III-548**

Solid sodium dithionate is added to a solution of cis chloroamminodiethylenediamino cobaltic chloride and a microcrystalline precipitate of cis chloroamminodiethylenediamino cobaltic dithionate precipitates. It may be recrystallized from hot water.

$$[ClNH_3Co(C_2H_8N_2)_2]Cl_2 + Na_2S_2O_6 \rightarrow [ClNH_3Co(C_2H_8N_2)_2]S_2O_6 + 2NaCl$$

A. Werner and R. Schmidt, Ann., **386**, 166 (1912) *20*

$[(C_3H_6O_2N)Co(C_2H_8N_2)_2]Cl_2$

$Na_2S_2O_6$ **III-549**

Excess solid sodium dithionate is added to a concentrated aqueous solution of sarcosine (methylaminoacetic acid) diethylenediamino cobaltic chloride and stirred to dissolve. Red prisms of sarcosine diethylenediamino cobaltic dithionate precipitate. They have one H_2O.

$$[(C_3H_6O_2N)Co(C_2H_8N_2)_2]Cl_2 + Na_2S_2O_6 \rightarrow [(C_3H_6O_2N)Co(C_2H_8N_2)_2]S_2O_6 + 2NaCl$$

J. Meisenheimer and E. Kinderlen, Ann., **438**, 270 (1924) *20*

$[Co(NH_3)_5NO_2]Cl_2$

$Na_3Co(NO_2)_6$ **III-550**

A solution of xanthocobalt chloride will react with a solution of sodium cobaltinitrite yielding a brown colored precipitate of xanthocobalt-cobaltic nitrite.

$$3\{[NO_2 \cdot Co(NH_3)_5]Cl_2\} + 2Na_3Co(NO_2)_6 \rightarrow \underline{[NO_2 \cdot Co(NH_3)_5]_3 \cdot [Co(NO_2)_6]_2} + 6NaCl$$

S. M. Jörgensen, Z. anorg. Chem., **5**, 147 (1894) *28*

$[Co(NH_3)_5(NO_2)]Cl_2$

Δ **III-551**

When mononitritopentammino cobaltic chloride is heated above 200°, decomposition occurs in the following manner.

$$2[Co(NH_3)_5(NO_2)]Cl_2 \rightarrow Co_2O_3 + 4NH_3 + 4NH_4Cl + 2N_2 + H_2O$$

G. L. Clark, A. J. Quick, and W. D. Harkens, J. Am. Chem. Soc., **42**, 2498 (1920) *16*

$[Copn_3]Cl_3$

HBr **III-552**

A concentrated aqueous solution of tripropylenediaminocobalt chloride is treated with concentrated hydrobromic acid. The precipitate is crystallized from warm water and tripropylenediaminocobalt bromide is obtained as yellow needles with three H_2O of crystallization.

$$[Co(C_3H_6(NH_2)_2)_3]Cl_3 + 3HBr \rightarrow [Co(C_3H_6(NH_2)_2)_3]Br_3 + 3HCl$$

P. Pfeiffer and T. Gassmann, Ann., **346**, 64 (1906) *20*

$[Co(NH_3)_2en_2]Cl_3$

HNO_3 **III-553**

An aqueous solution of diamminodiethylenediaminocobalt trichloride is treated with nitric acid. The precipitate formed is dissolved in water and precipitated by the addition of nitric acid. The precipitate is crystallized from water slightly acidified with nitric acid. Polyhedral crystals of diamminodiethylenediaminocobalt trinitrate with one H_2O separate. The compound is slightly soluble in water.

$$[Co(NH_3)_2(C_2H_8N_2)_2]Cl_3 + 3HNO_3 \rightarrow [Co(NH_3)_2(C_2H_8N_2)_2](NO_3)_3 + 3HCl$$

A. Werner, F. Braunlich and E. Rogowina, Ann., **351**, 76 (1907) *20*

$[Co(NH_3)_2en_2]Cl_3$

$HgCl_2$ **III-554**

A solution of diamminodiethylenediaminocobalt trichloride is treated with an aqueous solution of mercuric chloride and the precipitate crystallized from hot water. As the solution cools, golden yellow crystals of diamminodiethylenediaminocobalt chloromercurate deposit.

$$[Co(NH_3)_2(C_2H_8N_2)_2]Cl_3 + 5HgCl_2 \rightarrow [Co(NH_3)_2(C_2H_8N_2)_2]Cl_3 \cdot 5HgCl_2$$

A. Werner, F. Braunlich and E. Rogowina, Ann., **351**, 78 (1907) *20*

$[Copn_3]Cl_3$

KI **III-555**

An aqueous solution of tripropylenediaminocobaltchloride is treated with a solution of potassium iodide. The precipitate formed is crystallized from water and fine golden yellow crystals of tripropylenediaminocobalt iodide are obtained.

$$[Co(C_3H_6(NH_2)_2)_3]Cl_3 + 3KI \rightarrow \underline{[Co(C_3H_6(NH_2)_2)_3]I_3} + 3KCl$$

P. Pfeiffer and T. Gassmann, Ann., **346**, 64 (1906) *20*

$[Co(NH_3)_2en_2]Cl_3$

KI **III-556**

A warm aqueous solution of diethylenediaminodiamminocobalt trichloride is treated with the calculated amount of potassium iodide. An amorphous precipitate of diethylenediaminodiamminocobalt triiodide is formed.

$$[Co(NH_3)_2(C_2H_8N_2)_2]Cl_3 + 3KI \rightarrow \underline{[Co(NH_3)_2(C_2H_8N_2)_2]I_3} + 3KCl$$

A. Werner, F. Braunlich and E. Rogowina, Ann., **351**, 76 (1907) *20*

$[Co(NH_3)_2en_2]Cl_3$

KSCN **III-557**

An aqueous solution of diamminodiethylenediaminocobalt trichloride is treated with the calculated amount of potassium thiocyanate in solution. A voluminous precipitate forms which is crystallized from warm water and orange colored crystals of diamminodiethylenediaminocobalt trithiocyanate form.

$$[Co(NH_3)_2(C_2H_8N_2)_2]Cl_3 + 3KSCN \rightarrow [Co(NH_3)_2(C_2H_8N_2)_2](SCN)_3 + 3KCl$$

A. Werner, F. Braunlich and E. Rogowina, Ann., **351**, 77 (1907) *20*

$[Coen_3]Cl_3$

$K_3[Cr(CN)_6]$ **III-558**

1.25 Grams of triethylenediaminocobalt chloride are dissolved in water and to this solution is added a solution of 4 grams of chromium potassium cyanide. 1.2 Grams of a dark yellow crystalline mass precipitate. This is triethylenediaminocobalthexacyanochromate. The salt is insoluble in water.

$$[Co(C_2H_4(NH_2)_2)_3]Cl_3 + [Cr(CN)_6]K_3 \rightarrow [Co(C_2H_4(NH_2)_2)_3]\cdot[Cr(CN)_6] + 3KCl$$

P. Pfeiffer and H. Maismann, Ann., **346**, 75 (1906) *20*

$[Co(H_2O)_2en_2]Cl_3$

$Na_2S_2O_6$ **III-559**

5 Grams of cis diaquodiethylenediamino cobaltic chloride is dissolved in 12 cc of water, 25 cc of pyridine added and then 25 cc of a cold saturated aqueous solution of sodium dithionate. Bright violet crystals of hydroxoaquodiethylenediamino cobaltic dithionate deposit. These are washed with water and alcohol then dried with ether.

$$[(H_2O)_2Co(C_2H_8N_2)_2]Cl_3 + Na_2S_2O_6 \rightarrow [H_2OOHCo(C_2H_8N_2)_2]S_2O_6 + 2NaCl + HCl$$

A. Werner, Ann., **375**, 84 (1910) *20*

$[Co(NH_3)_2en_2]Cl_4 \cdot C_2O_4$

NH_4OH **III-560**

KI

Diamminodiethylenediaminocobaltic chloride oxalate is treated with a saturated water solution of ammonia at 0° and then heated in a bomb at 110° for two hours. The contents are then poured into water treated with KI and fractionally crystallized. The most insoluble fraction consists of brown crystals of diamminodiethylenediamine-cobaltic iodide oxalate.

$$[Co(NH_3)_2en_2]_2 \cdot Cl_4 \cdot C_2O_4 + 4KI \rightarrow [Co(NH_3)_2en_2]_2 \cdot I_4(C_2O_4) + 4KCl$$

A. Werner, Ann., **386**, 77 (1912) *41*

$[Co(NO_2)_2pn_2]I$

AgCl **III-561**

Trans dinitritoditrimethylenediamino cobaltic iodide is dissolved in warm water, cooled and freshly prepared silver chloride added. This is rubbed in a mortar, filtered and filtrate concentrated. Crystals of trans dinitroditrimethylenediamino cobaltic chloride deposit.

$$[(NO_2)_2Co(C_3H_{10}N_2)_2]I + AgCl \rightarrow [(NO_2)_2Co(C_3H_{10}N_2)_2]Cl + AgI$$

A. Werner and G. Lindenberg, Ann., **386**, 266 (1912) *20*

$[Co(NO_2)_2pn_2]I$

$AgNO_3$ **III-562**

Trans dinitritoditrimethylenediamino cobaltic iodide is dissolved in water and the calculated amount of silver nitrate in solution added. The solution is filtered, the filtrate concentrated and then allowed to stand overnight. The precipitate which forms is recrystallized from hot water. Crystals of trans dinitritoditrimethylenediamino cobaltic nitrate deposit.

$$[(NO_2)_2Co(C_3H_{10}N_2)_2]I + AgNO_3 \rightarrow [(NO_2)_2Co(C_3H_{10}N_2)_2]NO_3 + AgI$$

A. Werner and G. Lindenberg, Ann., **386**, 269 (1912) *20*

$[Co(NO_2)_2en_2]I$

Ag_2O **III-563**

H_2SO_4

Trans dinitritodiethylenediamino cobaltic iodide is dissolved in water and shaken with silver oxide. Trans dinitritodiethylenediamino cobaltic hydroxide is formed and this is converted to the bisulfate by neutralizing with sulfuric acid. Alcohol is added to the solution and it separates into two layers. The deep colored layer is treated with additional sulfuric acid and it sets to a crystalline mass. This is recrystallized from dilute alcohol and yellow brown needles of trans nitritodiethylenediamino cobaltic bisulfate separate.

(a) $$Ag_2O + H_2O \rightarrow 2AgOH$$

(b) $$[(NO_2)_2Co(C_2H_8N_2)_2]I + AgOH \rightarrow [(NO_2)_2Co(C_2H_8N_2)_2]OH + AgI$$

(c) $$[(NO_2)_2Co(C_2H_8N_2)_2]OH + H_2SO_4 \rightarrow [(NO_2)_2Co(C_2H_8N_2)_2]HSO_4 + H_2O$$

A. Werner and L. Cohn, Ann., **386**, 250 (1912) *20*

$[CoCO_3en_2]I$

Ag_2SO_4 **III-564**

A cold saturated aqueous solution of carbonato diethylene diamino-cobaltic iodide is treated with a concentrated aqueous solution of silver sulfate. The solution is filtered and concentrated on the water bath. Upon cooling, reddish black prismatic crystals of carbonatodiethylenediamino cobaltic sulfate with five H_2O deposit. The water of crystallization is lost at 100°C.

$$2[CO_3Co(C_2H_8N_2)_2]I + Ag_2SO_4 \rightarrow [CO_3Co(C_2H_8N_2)_2]_2SO_4 + 2AgI$$

A. Werner and J. Rapiport, Ann., **386**, 76 (1912) *20*
41

$[CoNH_3NO_2en_2]I_2$

$Na_2S_2O_6$ **III-565**

1 Gram of trans nitritoamminodiethylenediamino cobaltic iodide is dissolved in 20 cc of water and a concentrated aqueous solution of sodium dithionate added dropwise until crystallization begins. Golden yellow lustrous needles of trans nitritoamminodiethylenediamine cobaltic dithionate deposit.

$$[NO_2NH_3Co(C_2H_8N_2)_2]I_2 + Na_2S_2O_6 \rightarrow [NO_2NH_3Co(C_2H_8N_2)_2]S_2O_6 + 2NaI$$

A. Werner and W. Boes, Ann., **386**, 227 (1912) *20*

$[Co(NH_3)_2en_2]I_3$

$AgNO_3$ **III-566**

HCl

$CoCl_2$

2 Grams of diamminodiethylenediaminocobalt triiodide are dissolved in 7 cc of water and the calculated amount of silver nitrate to precipitate the iodine is added. The solution is filtered to remove the silver iodide and hydrochloric acid added to the filtrate to remove any excess silver. The solution is again filtered and the filtrate saturated with cobaltous chloride. The color of the solution changes from bright red to brownish red. Concentrated hydrochloric acid is added until the color of the solution changes to blue green, and bright green crystals of the double compound diamminodiethylenediaminocobalt trichloride chlorocobaltate form.

(a) $[Co(NH_3)_2(C_2H_8N_2)_2]I_3 + 3AgNO_3 \rightarrow [Co(NH_3)_2(C_2H_8N_2)_2](NO_3)_3 + \underline{3AgI}$

(b) $[Co(NH_3)_2(C_2H_8N_2)_2](NO_3)_3 + 3HCl \rightarrow [Co(NH_3)_2(C_2H_8N_2)_2]Cl_3 + 3HNO_3$

(c) $[Co(NH_3)_2(C_2H_8N_2)_2]Cl_3 + CoCl_2 \rightarrow [Co(NH_3)_2(C_2H_8N_2)_2]Cl_3 \cdot CoCl_2$

A. Werner and C. Kreutzer, Ann., **351**, 83 (1907) *20*

$[Co(NH_3)_2en_2]I_3$

Ag_2O **III-567**

HBr

2 Grams of diamminodiethylenediaminocobalt triiodide are dissolved in 15 cc of water and silver oxide added, while agitating constantly, until no more silver iodide precipitates. The solution is filtered and saturated with hydrobromic acid at water bath temperature. The solution is placed in a desiccator over sulfuric acid to crystallize. Orange crystals of diamminodiethylenediaminocobalt tribromide form.

(a) $Ag_2O + H_2O \rightarrow 2AgOH$

(b) $[Co(NH_3)_2(C_2H_8N_2)_2]I_3 + 3AgOH$
$\rightarrow [Co(NH_3)_2(C_2H_8N_2)_2](OH)_3 + 3\underline{AgI}$

(c) $[Co(NH_3)_2(C_2H_8N_2)_2](OH)_3 + 3HBr$
$\rightarrow [Co(NH_3)_2(C_2H_8N_2)_2]Br_3 + 3H_2O$

A. Werner and C. Kreutzer, Ann., **351**, 83 (1907) *20*

$[Copn_3]I_3$

KSCN **III-568**

2 Grams of pure tripropylenediamminocobalt iodide are dissolved in a concentrated solution and treated with an aqueous solution of potassium thiocyanate. Tripropylenediaminocobalt thiocyanate precipitates as fine yellow crystals.

$[Co(C_3H_6(NH_2)_2)_3]I_3 + 3KSCN$
$\rightarrow [Co(C_3H_6(NH_2)_2)_3]\cdot[(SCN)_3] + 3KI$

P. Pfeiffer and T. Gassmann, Ann., **346**, 66 (1906) *20*

$[Copn_3]I_3$

$K_3[Co(CN)_6]$ **III-569**

A concentrated aqueous solution of tripropylenediaminocobalt iodide is treated with a concentrated solution of potassium cobalt cyanide and a yellow precipitate forms. It is crystallized from hot water and tripropylenediaminocobaltohexacyanocobaltate is obtained.

$[Co(C_3H_6(NH_2)_2)_3]I_3 + [Co(CN)_6]K_3$
$\rightarrow [Co(C_3H_6(NH_2)_2)_3]\cdot[Co(CN)_6] + 3KI$

P. Pfeiffer and T. Gassmann, Ann., **346**, 67 (1906) *20*

$[Copn_3]I_3$

$K_3[Cr(CN)_6]$ **III-570**

A solution of tripropylenediaminocobalt iodide in water is treated with one of chromium potassium cyanide. Small golden yellow crystals of tripropylenediaminocobalthexacyanochromate with $3H_2O$ separate. They are insoluble in water and are not decomposed at 140°C.

$$[Co(C_3H_6(NH_2)_2)_3]I_3 + [Cr(CN)_6]K_3 \rightarrow [Co(C_3H_6(NH_2)_2)_3]\cdot[Cr(CN)_6] + 3KI$$

P. Pfeiffer and H. Maismann, Ann., **346**, 77 (1906) *20*

$[Copn_3]I_3$

$K_3[Cr(C_2O_4)_3]$ **III-571**

Tripropylenediaminocobalt iodide and blue chromium potassium oxalate are dissolved in water in molecular proportions. A chocolate colored precipitate which soon changes to fine needles forms. This is tripropylenediaminocobalttrioxalatochromate.

$$[Co(C_3H_6(NH_2)_2)_3]I_3 + [Cr(C_2O_4)_3]K_3 \rightarrow [Co(C_3H_6(NH_2)_2)_3]\cdot[Cr(C_2O_4)_3] + 3KI$$

P. Pfeiffer and T. Gassmann, Ann., **346**, 68 (1906) *20*

$[Copn_3]I_3$

$K_3Fe(CN)_6$ **III-572**

An aqueous solution of tripropylenediaminocobalt iodide is mixed with one of potassium ferricyanide. Red yellow needles of tripropylenediaminocobalthexacyanoferrate with three H_2O form. They are insoluble in water.

$$[Co(C_3H_6(NH_2)_2)_3]I_3 + K_3Fe(CN)_3 \rightarrow [Co(C_3H_6(NH_2)_2)_3]\cdot Fe(CN)_6 + 3KI$$

P. Pfeiffer and T. Gassmann, Ann., **346**, 68 (1906) *20*

$[Co(NO_2)_2pn_2]NO_2$

KBr **III-573**

Solid potassium bromide is added to a concentrated aqueous solution of trans dinitritoditrimethylenediamino cobaltic nitrite and a yellow precipitate of trans dinitroditrimethylenediamino cobaltic bromide forms. It is recrystallized from hot water and obtained as brownish yellow crystals with one H_2O.

$$[(NO_2)_2Co(C_3H_{10}N_2)_2]NO_2 + KBr \rightarrow [(NO_2)_2Co(C_3H_{10}N_2)_2]Br + KNO_2$$

A. Werner and G. Lindenberg, Ann., **386**, 267 (1912) *20*

$[Co(NO_2)_2pn_2]NO_2$

KI **III-574**

3 Grams of trans dinitritoditrimethylenediamino cobaltic nitrite are dissolved in the minimum amount of water and solid potassium iodide added. A yellow lustrous precipitate forms which is recrystallized from hot water and rhombic prisms of trans dinitritoditrimethylenediamino cobaltic iodide with two H_2O deposit.

$$[(NO_2)_2Co(C_3H_{10}N_2)_2]NO_2 + KI \rightarrow [(NO_2)_2Co(C_3H_{10}N_2)_2]I + KNO_2$$

A. Werner and G. Lindenberg, Ann., **386**, 268 (1912) *20*

$[CoNO_2NCSen_2]NO_2$

KSCN **III-575**

A concentrated aqueous solution of trans nitritoisothiocyanatodiethylenediamino cobaltic nitrite is treated with solid potassium thiocyanate and red needles of trans nitritoisothiocyanatodiethylenediamino cobaltic thiocyanate precipitate. The salt may be purified by reprecipitation from aqueous solution by the addition of potassium thiocyanate.

$$[NO_2NCSCo(C_2H_8N_2)_2]NO_2 + KSCN \rightarrow [NO_2NCSCo(C_2H_8N_2)_2]SCN + KNO_2$$

A. Werner and R. Schmidt, Ann., **386**, 163 (1912) *20*

$[Co(NO_2)_2en_2]NO_2$

Δ **III-576**

An aqueous solution of pure cis dinitritodiethylenediamino cobaltic nitrite is heated to boiling and kept at that temperature for 6 hours during which time steam is passed into the solution. Upon cooling, crystals of the trans isomer, dinitritodiethylenediamino cobaltic nitrite deposit.

$$(cis)[(NO_2)_2Co(C_2H_8N_2)_2]NO_2 + \Delta \rightarrow (trans)[(NO_2)_2Co(C_2H_8N_2)]\cdot NO_2$$

A. Werner and L. Cohn, Ann., **386**, 249 (1912) *20*

$[CoNH_3Clen_2](NO_2)_2$

$AgNO_2$ **III-577**

Silver nitrite reacts with cis chloroamminodiethylenediamino cobaltic nitrite to form cis nitritoamminodiethylenediamino cobaltic nitrite.

$$[ClNH_3Co(C_2H_8N_2)_2](NO_2)_2 + AgNO_2 \rightarrow [NO_2NH_3Co(C_2H_8N_2)_2](NO_2)_2 + AgCl$$

A. Werner and W. Boes, Ann., **386**, 217 (1912) *20*

$[CoNH_3Clen_2](NO_2)_2$

$AgNO_2$ **III-578**

$Na_2S_2O_6$

The mother liquor obtained by reacting cis chloroamminodiethylenediamino cobaltic nitrite with silver nitrite and sodium dithionate as described in reaction III-533 and filtering off the cis nitritoamminodiethylenediamino cobaltic dithionate contains trans nitritoamminodiethylenediamino cobaltic dithionate and it may be recovered by evaporation of the solvent.

(a) $[ClNH_3Co(C_2H_8N_2)_2](NO_2)_2 + AgNO_2 \rightarrow [NO_2NH_3Co(C_2H_8N_2)_2](NO_2)_2 + AgCl$

(b) $[NO_2NH_3Co(C_2H_8N_2)_2](NO_2)_2 + Na_2S_2O_6 \rightarrow [NO_2NH_3Co(C_2H_8N_2)_2]S_2O_6 + 2NaNO_2$

A. Werner and R. Schmidt, Ann., **386**, 168 (1912) *20*

$[CoNH_3Clen_2](NO_2)_2$

Decomp. **III-579**

Ammonia is evolved from a concentrated aqueous solution of trans chloroamminodiethylenediamino cobaltic nitrite and trans chloronitritodiethylenediamino cobaltic nitrite is formed.

$$[ClNH_3Co(C_2H_8N_2)_2](NO_2)_2 \rightarrow [ClNO_2Co(C_2H_8N_2)_2]NO_2 + NH_3$$

A. Werner and R. Schmidt, Ann., **386**, 175 (1912) *20*

$[CoNO_2NCSen_2]NO_3$

$AgNO_3$ **III-580**

Trans nitritoisothiocyanatodiethylenediamino cobaltic nitrate is dissolved in a little water and a solution of the calculated quantity of silver nitrate added. A bright yellow curdy precipitate forms which is recrystallized from hot water and long yellow needles of trans silver isothiocyanatonitritodiethylenediamino cobaltic nitrate are deposited.

$$[NO_2NCSCo(C_2H_8N_2)_2]NO_3 + AgNO_3 \rightarrow [AgNCSNO_2Co(C_2H_8N_2)_2](NO_3)_2$$

A. Werner and N. Gosling, Ann., **386**, 245 (1912) *20*

$[Co(NH_3)_3C_2O_4H_2O]NO_3$

HBr **III-581**

5 Grams of oxalatoaquotriamminocobaltic nitrate are dissolved in 110 cc of water, cooled and 80 cc of concentrated hydrobromic acid added. Oxalatoaquotriamminocobaltic bromide precipitates and it is purified by dissolving in water and reprecipitating with hydrobromic acid. The precipitate is washed with alcohol and ether. The compound is deposited as bluish red prisms and the yield is 3.4 grams. The compound crystallizes with one H_2O.

$$[(C_2O_4)H_2OCo(NH_3)_3]NO_3 + HBr \rightarrow [(C_2O_4)H_2OCo(NH_3)_3]Br + HNO_3$$

A. Werner, E. Bindschedler and C. Sackur, Ann., **405**, 231 (1914) *20*

$[Co(NH_3)_3C_2O_4H_2O]NO_3$

HCl **III-582**

5 Grams of oxalatoaquotriamminocobaltic nitrate are dissolved in 110 cc of warm water, cooled in an ice bath, filtered and 20 cc of concentrated hydrochloric acid added. A red crystalline precipitate forms almost immediately. 30 cc hydrochloric acid is added, the solution allowed to stand 30 minutes and an additional 50 cc of hydrochloric acid added. The precipitate is filtered off, washed with half concentrated hydrochloric acid then alcohol. It is then recrystallized from water by dissolving 3.8 grams in 100 cc of water and adding an equal volume of concentrated hydrochloric acid. Oxalatoaquotriamminocobaltic chloride is deposited as a violet red crystalline powder. Yield about 3 grams.

$$[(C_2O_4)H_2OCo(NH_3)_3]NO_3 + HCl \rightarrow [(C_2O_4)H_2OCo(NH_3)_3]Cl + HNO_3$$

A. Werner, E. Bindschedler and C. Sackur, Ann., **405**, 229 (1914) *20*

$[Co(NO_2)_2en_2]NO_3$

HNO_3 **III-583**

KSCN

10 Grams of cis dinitritodiethylenediamino cobaltic nitrate are added to 60 cc of dilute nitric acid (30 cc concentrated nitric acid and 30 cc water) and warmed on the water bath to dissolve. The solution is allowed to stand overnight and nitronitratodiethylenediamino cobaltic nitrate precipitates.

5 Grams of this salt are dissolved in 20 cc of water and 3.5 grams of potassium thiocyanate added. Trans nitritoisothiocyanatodiethylenediamino cobaltic thiocyanate is formed.

(a) $$[(NO_2)_2Co(C_2H_8N_2)_2]NO_3 + HNO_3 \rightarrow [NO_3NO_2Co(C_2H_8N_2)_2]NO_3 + HNO_2$$

(b) $$[NO_3NO_2Co(C_2H_8N_2)_2]NO_3 + 2KSCN \rightarrow [NO_2NCSCo(C_2H_8N_2)_2]SCN + 2KNO_3$$

A. Werner and N. Gosling, Ann., **386**, 241 (1912) *20*

$[Co(NO_2)_2en_2]NO_3$

HNO_3 **III-584**

100 Grams of cis dinitritodiethylenediamino cobaltic nitrate are dissolved in 200 cc of water and an equal volume of concentrated nitric acid is added. This solution is heated for 4 hours then cooled. The solution is filtered, the filtrate concentrated and nitric acid added when an additional quantity of the reaction product is obtained. The combined residues are recrystallized from hot water and trans nitritonitratodiethylenediamino cobaltic nitrate is deposited as chamois colored small lustrous crystals.

$$[(NO_2)_2Co(C_2H_8N_2)_2]NO_3 + HNO_3 \rightarrow [NO_2NO_3Co(C_2H_8N_2)_2]NO_3 + HNO_2$$

A. Werner and L. Cohn, Ann., **386**, 252 (1912) *20*

$[Co(NH_3)_4(NO_2)_2]NO_3$

$KAuCl_4$ **III-585**

Brown colored crystals of flavo cobalt chloroaurate are obtained when a solution of flavo cobalt nitrate reacts with a solution of potassium chloroaurate.

$$[(NO_2)\cdot Co(NH_3)_4]\cdot NO_3 + KAuCl_4 \rightarrow \underline{[(NO_2)\cdot Co(NH_3)_4]\cdot AuCl_4} + KNO_3$$

S. M. Jörgensen, Z. anorg. Chem., **5**, 147 (1894) *28*

$[Co(NH_3)_4(NO_2)_2]NO_3$

$K[(NH_3)_2Co(NO_2)_4]$ **III-586**

A solution of flavo cobalt nitrate will react with a solution of potassium diamminecobalt nitrite yielding a yellow precipitate.

$$[(NO_2)_2\cdot Co(NH_3)_4]\cdot NO_3 + K[(NH_3)_2\cdot Co\cdot(NO_2)_4] \rightarrow \underline{(NO_2)_2\cdot Co(NH_3)_4\cdot(NO_2)_2\cdot(NH_3)_2\cdot Co(NO_2)_2} + KNO_3$$

S. M. Jörgensen, Z. anorg. Chem., **5**, 147 (1894) *28*

$[CoClNO_2en_2]NO_3$

KSCN **III-587**

Solid potassium thiocyanate is added to a solution of 5 grams of trans chloronitritodiethylenediamino cobaltic nitrate in 120 cc of water. A brown crystalline precipitate forms. It is recrystallized from water by dissolving 2 grams in 40 cc. Trans nitritoisothiocyanatodiethylenediamino cobaltic thiocyanate deposits as brown prismatic crystals.

$$[ClNO_2Co(C_2H_8N_2)_2]NO_3 + 2KSCN \rightarrow [NO_2NCSCo(C_2H_8N_2)_2]SCN + KCl + KNO_3$$

A. Werner and N. Gosling, Ann., **386**, 240 (1912) *20*

$[Co(NH_3)_3C_2O_4H_2O]NO_3$

KSCN **III-588**

40 Grams of potassium thiocyanate are added to a warm aqueous solution of 4 grams of oxalatoaquotriamminocobaltic nitrate in 20 cc of water. The solution is heated until all solids are dissolved, then filtered. After a short time, rhombic crystals of cobaltic thiocyanatooxalatotriammine deposit.

$$[(C_2O_4)H_2OCo(NH_3)_3]NO_3 + KSCN \rightarrow [(C_2O_4)CoSCN(NH_3)_3] + KNO_3 + H_2O$$

A. Werner, E. Bindschedler and C. Sackur, Ann., **405**, 236 (1914) *20*

$[Co(NO_2)_2en_2]NO_3$

KSCN **III-589**

2 Grams of cis dinitritodiethylenediamino cobaltic nitrate are dissolved in 20 cc of water and 6 grams of potassium thiocyanate added. Cis dinitritodiethylenediamino cobaltic thiocyanate precipitates. It is recrystallized from hot water and the compound is obtained as lustrous yellowish brown tablets.

$$[(NO_2)_2Co(C_2H_8N_2)_2]NO_3 + KSCN \rightarrow [(NO_2)_2Co(C_2H_8N_2)_2]SCN + KNO_3$$

A. Werner and L. Cohn, Ann., **386**, 249 (1912) *20*

$[Co(NO_2)_2en_2]NO_3$

KSCN **III-590**

12 Grams of trans dinitritodiethylenediamino cobaltic nitrate are dissolved in 150 cc of warm water and 9 grams of solid potassium thiocyanate added. Trans nitritoisothiocyanatodiethylenediamino cobaltic thiocyanate precipitates.

$$[(NO_2)_2Co(C_2H_8N_2)_2]NO_3 + 2KSCN \rightarrow \underline{[NO_2NCSCo(C_2H_8N_2)_2]SCN} + KNO_2 + KNO_3$$

A. Werner and N. Gosling, Ann., **386**, 241 (1912) *20*

$[Co(NH_3)_4(NO_2)_2]NO_3$

$K_2Cr_2O_7$ **III-591**

Orange-yellow crystals of flavo cobalt dichromate are obtained when a concentrated solution of flavo cobalt nitrate reacts with a saturated solution of potassium dichromate.

$$2[(NO_2)_2 \cdot Co \cdot (NH_3)_4]NO_3 + K_2Cr_2O_7 \rightarrow \underline{[(NO_2)_2 \cdot Co \cdot (NH_3)_4]_2 \cdot Cr_2O_7} + 2KNO_3$$

S. M. Jörgensen, Z. anorg. Chem., **5**, 147 (1894) *28*

$[Co(NH_3)_4(NO_2)_2]NO_3$

K_2PtCl_4 **III-592**

Brown crystals of flavo cobalt chloroplatinite are obtained when a solution of flavo cobalt nitrate reacts with a solution of potassium chloroplatinite.

$$2[(NO_2)_2 \cdot Co \cdot (NH_3)_4] \cdot NO_3 + K_2PtCl_4 \rightarrow \underline{[(NO_2)_2 \cdot Co \cdot (NH_3)_4]_2 \cdot PtCl_4} + 2KNO_3$$

S. M. Jörgensen, Z. anorg. Chem., **5**, 147 (1894) *28*

$[Co(NH_3)_3C_2O_4H_2O]NO_3$

NH_3 **III-593**

1 Gram of oxalatoaquotriamminocobaltic nitrate is dissolved in 20 cc of warm water, cooled and 15 cc of 25% ammonia added. A bluish red precipitate forms which is filtered off, washed with water and alcohol then dried over sulfuric acid. The compound formed is cobalt hydroxooxalatotriammine. It is insoluble in water, alcohol and ether as well as dilute acetic acid.

$$[(C_2O_4)H_2OCo(NH_3)_3]NO_3 + NH_3 \rightarrow \underline{[(C_2O_4)OHCo(NH_3)_3]} + NH_4NO_3$$

A. Werner, E. Bindschedler and C. Sackur, Ann., **405**, 227 (1914) *20*

$[CoNO_2NO_3en_2]NO_3$

$(NH_4)_2SO_4$

III-594

4 Grams of trans nitritonitratodiethylenediamino cobaltic nitrate are dissolved in 10 cc of hot water, the solution filtered and cooled. Solid ammonium sulfate is added in the ratio of 0.7 gram for each gram of the nitrate. After the ammonium sulfate has dissolved, alcohol is added until the precipitate just fails to redissolve. A bright yellow precipitate forms which is recrystallized from 50% alcohol and bright orange needles of trans nitritoaquodiethylenediamino cobaltic sulfate deposit.

$$2[NO_2NO_3Co(C_2H_8N_2)_2]NO_3 + (NH_4)_2SO_4 + H_2O \rightarrow [NO_2H_2OCo(C_2H_8N_2)_2]_2SO_4 + 2NH_4NO_3$$

A. Werner and L. Cohn, Ann., **386**, 250 (1912) *20*

$[Co(NH_3)_3C_2O_4H_2O]NO_3$

$(NH_4)_2SO_4$

III-595

8 Grams of oxalatoaquotriamminocobaltic nitrate are dissolved in 160 cc of water and a solution of 10 grams of ammonium sulfate in 40 cc of water added. A crystalline precipitate forms. It is washed with cold water and dried. Yield 7 grams of oxalatoaquotriamminocobaltic sulfate. It is purified by recrystallization from water acidified with acetic acid. The crystals have one H_2O.

$$2[(C_2O_4)H_2OCo(NH_3)_3]NO_3 + (NH_4)_2SO_4 \rightarrow [(C_2O_4)H_2OCo(NH_3)_3]_2SO_4 + 2NH_4NO_3$$

A. Werner, E. Bindschedler and C. Sackur, Ann., **405**, 233 (1914) *20*

$[Co(NH_3)_4(NO_2)_2]NO_3$

$(NH_4)_2SO_4$

III-596

Yellow crystals of flavo cobalt sulfate are obtained when a solution of flavo cobalt nitrate reacts with a diluted solution of ammonium sulfate.

$$2[(NO_2)_2\cdot Co(NH_3)_4]\cdot NO_3 + (NH_4)_2SO_4 \rightarrow \underline{[(NO_2)_2\cdot Co(NH_3)_4]_2\cdot SO_4} + 2NH_4NO_3$$

S. M. Jörgensen, Z. anorg. Chem., **5**, 147 (1894) *28*

$[Co(NH_3)_4(NO_2)_2]NO_3$

$NaNO_2$ **III-597**

$C_2H_4O_2$

A solution of flavo cobalt nitrate will react with sodium nitrite and acetic acid at boiling temperature yielding a yellow precipitate.

$$2[(NO_2)_2 \cdot Co(NH_3)_4]NO_3 + 4NaNO_2 + 2C_2H_4O_2 \rightarrow \underline{(NO_2)_2 \cdot Co \cdot (NH_3)_4 \cdot (NO_2)_2 \cdot (NH_3)_2 \cdot Co(NO_2)_2} + 2NaNO_3 + 2NaC_2H_3O_2 + N_2 + 4H_2O$$

S. M. Jörgensen, Z. anorg. Chem., **5**, 147 (1894) *28*

$[Co(NH_3)_3C_2O_4H_2O]NO_3$

$Na_2S_2O_6$ **III-598**

3 Grams of oxalatoaquotriamminocobaltic nitrate are dissolved in 60 cc of water, cooled and a solution of 8 grams of sodium dithionate in 45 cc of water added. The precipitate which forms is washed with cold water in which it is practically insoluble. It is recrystallized from water by dissolving 2.5 grams in 200 cc of hot water containing acetic acid, filtering and allowing to cool slowly. Bordeaux red crystals of oxalatoaquotriamminocobaltic dithionate are formed.

$$2[(C_2O_4)H_2OCo(NH_3)_3]NO_3 + Na_2S_2O_6 \rightarrow [(C_2O_4)H_2OCo(NH_3)_3]_2S_2O_6 + 2NaNO_3$$

A. Werner, E. Bindschedler and C. Sackur, Ann., **405**, 232 (1914) *20*

$[Co(NH_3)_4(NO_2)_2]NO_3$

$Na_3Co(NO_2)_6$ **III-599**

A solution of flavo cobalt nitrate will react with a solution of sodium cobaltinitrite yielding yellow colored crystals.

$$3[(NO_2)_2 \cdot Co(NH_3)_4]NO_3 + Na_3Co(NO_2)_6 \rightarrow \underline{[(NO_2)_2 \cdot Co(NH_3)_4]_3 \cdot (NO_2)_6 \cdot Co} + 3NaNO_3$$

S. M. Jörgensen, Z. anorg. Chem., **5**, 147 (1894) *28*

$[CoNH_3Clen_2](NO_3)_2$

$AgNO_2$

$Na_2S_2O_6$ **III-600**

A dilute solution of trans chloroamminodiethylenediamino cobaltic nitrate is treated with 2 moles of silver nitrite, the solution heated until it becomes a pure yellow color, filtered and solid sodium dithionate added to the filtrate. A mixture of the cis and trans nitritoamminodiethylenediamino cobaltic nitrite is precipitated. These two isomers may be separated by fractional solution. The trans salt is more soluble than the cis and the trans may be precipitated from its solution as large prisms. The cis form is obtained by further addition of sodium dithionate.

(a) $[ClNH_3Co(C_2H_8N_2)_2](NO_3)_2 + 3AgNO_2$
$\rightarrow [NO_2NH_3Co(C_2H_8N_2)_2](NO_2)_2 + \underline{AgCl} + 2AgNO_3$

(b) $[NO_2NH_3Co(C_2H_8N_2)_2](NO_2)_2 + Na_2S_2O_6$
$\rightarrow [NO_2NH_3Co(C_2H_8N_2)_2]S_2O_6 + 2NaNO_2$

A. Werner and R. Schmidt, Ann., **386**, 174 (1912) *20*

$[CoH_2ONCSen_2](NO_3)_2$

$AgNO_3$ **III-601**

An excess of a concentrated aqueous solution of silver nitrate is added to a cold concentrated aqueous solution of trans aquoisothiocyanatodiethylenediamino cobaltic nitrate. The color of the solution changes from violet to bright red and bright red needles of the silver nitrate double salt precipitate. The compound may be recrystallized from cold water by the addition of silver nitrate solution.

$[H_2ONCSCo(C_2H_8N_2)_2](NO_3)_2 + 2AgNO_3$
$\rightarrow [Ag_2SCNH_2OCo(C_2H_8N_2)_2](NO_3)_4$

A. Werner and R. Schmidt, Ann., **386**, 163 (1912) *20*

$[Co(NH_3)_5SCN](NO_3)_2$

$AgNO_3$ **III-602**

When silver nitrate is added to a solution of isothiocyanatopentammino cobaltic nitrate, an addition compound precipitates out as yellow leaflets. No silver can be precipitated from this compound by addition of hydrochloric acid.

$[SCNCo(NH_3)_5](NO_3)_2 + AgNO_3 \rightarrow \underline{[AgSCNCo(NH_3)_5](NO_3)_3}$

T. S. Price and S. A. Brazier, J. Chem. Soc. (London), **107**, 1716 (1915) *57*

$[CoNH_3H_2Oen_2](NO_3)_2$

HNO_3 **III-603**

$Na_2S_2O_6$

Trans aquoamminodiethylenediamino cobaltic nitrate is added to concentrated nitric acid and the solution evaporated to dryness on the water bath. The residue is a mixture of cis and trans nitratoamminodiethylenediamino cobaltic nitrate with the trans form predominating. The residue is dissolved in water and solid sodium dithionate added. The cis isomer precipitates as an orange colored powder. This is filtered off and the filtrate allowed to crystallize. After several hours the trans form deposits as orange needles.

(a) $[H_2ONH_3Co(C_2H_8N_2)_2](NO_3)_3$
$\rightarrow [NO_3NH_3Co(C_2H_8N_2)_2](NO_3)_2 + H_2O$

(b) $[NO_3NH_3Co(C_2H_8N_2)_2](NO_3)_2 + Na_2S_2O_6$
$\rightarrow [NO_3NH_3Co(C_2H_8N_2)_2]S_2O_6 + 2NaNO_3$

A. Werner and C. Rix, Ann., **386**, 215 (1912) *20*

$[CoNH_3NO_2en_2](NO_3)_2$

KBr **III-604**

Solid potassium bromide is added to a concentrated aqueous solution of trans nitritoamminodiethylenediamino cobaltic nitrate and a brownish yellow precipitate forms. It is recrystallized from hot water and trans nitritoamminodiethylenediamino cobaltic bromide is obtained as short dark brown thick crystals.

$[NO_2NH_3Co(C_2H_8N_2)_2](NO_3)_2 + 2KBr$
$\rightarrow [NO_2NH_3Co(C_2H_8N_2)_2]Br_2 + 2KNO_3$

A. Werner and W. Boes, Ann., **386**, 226 (1912) *20*

$[CoNH_3NO_2en_2](NO_3)_2$

KI **III-605**

2.3 Grams of trans nitritoamminodiethylenediamino cobaltic nitrate are dissolved in 7 cc of water and 4 grams of potassium iodide added. A yellow crystalline powder precipitates. This is removed by filtration, dissolved in 12 cc of hot water and allowed to crystallize. Trans nitritoamminodiethylenediamino cobaltic iodide deposits as lustrous long black prisms with one H_2O.

$[NO_2NH_3Co(C_2H_8N_2)_2](NO_3)_2 + 2KI$
$\rightarrow [NO_2NH_3Co(C_2H_8N_2)_2]I_2 + 2KNO_3$

A. Werner and W. Boes, Ann., **386**, 225 (1912) *20*

$[CoNH_3NO_2en_2](NO_3)_2$

KSCN **III-606**

5 Grams of trans nitritoamminodiethylenediamino cobaltic nitrate are dissolved in 20 cc of cold water and 5 grams of solid potassium thiocyanate added. After a short time a yellow crystalline precipitate forms. It is dissolved in 15 cc of warm water acidified with acetic acid and lustrous brownish yellow thick plates of trans nitritoamminodiethylenediamino cobaltic thiocyanate form as the solution cools.

$$[NO_2NH_3Co(C_2H_8N_2)_2](NO_3)_2 + 2KSCN \rightarrow [NO_2NH_3Co(C_2H_8N_2)_2](SCN)_2 + 2KNO_3$$

A. Werner and W. Boes, Ann., **386**, 226 (1912) *20*

$[CoNH_3NO_2en_2](NO_3)_2$

KSCN **III-607**

HCl

5 Grams of trans nitritoamminodiethylenediamino cobaltic nitrate are dissolved in 20 cc of water, 5 grams potassium thiocyanate and 5 drops of 10% potassium hydroxide added and the solution heated until the odor of ammonia disappears. Upon cooling, crystals of trans nitritoisothiocyanatodiethylenediamino cobaltic thiocyanate and trans diisothiocyanatodiethylenediamino cobaltic thiocyanate deposit. These crystals are removed by filtration triturated with hydrochloric acid and allowed to stand some time and the difficultly soluble trans diisothiocyanatodiethylenediamino cobaltic chloride removed by filtration. Addition of alcohol to the filtrate causes the precipitation of trans nitritoisothiocyanatodiethylenediamino chloride as needle crystals. This chloride is dissolved in water and solid potassium thiocyanate added. Trans nitritoisothiocyanatodiethylenediamino cobaltic thiocyanate precipitates and is recrystallized from hot water.

(a) $$2[NO_2NH_3Co(C_2H_8N_2)_2](NO_3)_2 + 5KSCN \rightarrow [NO_2NCSCo(C_2H_8N_2)_2]SCN + [(NCS)_2Co(C_2H_8N_2)_2]SCN + 4KNO_3 + KNO_2 + 2NH_3$$

(b) $$[(NCS)_2Co(C_2H_8N_2)_2]SCN + HCl \rightarrow [(NCS)_2Co(C_2H_8N_2)_2]Cl + HSCN$$

A. Werner and N. Gosling, Ann., **386**, 242 (1912) *20*

$[CoH_2OBren_2](NO_3)_2$

NaBr **III-608**

10 Grams of cis bromoaquodiethylenediamino cobaltic nitrate are dissolved in 20 cc of water with the aid of heat. The solution is cooled, filtered and solid sodium bromide added to the filtrate until precipitation is complete. Blue violet crystals of cis bromoaquodiethylenediamino cobaltic bromide deposit. The yield is 5.5 grams.

$$[BrH_2OCo(C_2H_8N_2)_2](NO_3)_2 + 2NaBr \rightarrow [BrH_2OCo(C_2H_8N_2)_2]Br_2 + 2NaNO_3$$

A. Werner and R. Schmidt, Ann., **386**, 127 (1912) *20*

$[CoH_2ONCSen_2](NO_3)_2$

$NaNO_2$ **III-609**

A cold saturated aqueous solution of trans aquoisothiocyanatodiethylenediamino cobaltic nitrate is treated with solid sodium nitrite and trans aquoisothiocyanatodiethylenediamino cobaltic nitrite precipitates as a dark violet red powder. The compound is fairly stable.

$$[H_2ONCSCo(C_2H_8N_2)_2](NO_3)_2 + 2NaNO_2 \rightarrow \underline{[H_2ONCSCo(C_2H_8N_2)_2](NO_2)_2} + 2NaNO_3$$

A. Werner and R. Schmidt, Ann., **386**, 161 (1912) *20*

$[CoH_2ONCSen_2](NO_3)_2$

$NaNO_2$ **III-610**

A concentrated aqueous solution of trans aquoisothiocyanatodiethylenediamino cobaltic nitrate is acidified slightly with acetic acid and solid sodium nitrite added while stirring until the color of the solution becomes bright red and a bright red precipitate forms. The precipitate is removed, dissolved in cold water and crystals of trans nitritoisothiocyanatodiethylenediamino cobaltic nitrite are reprecipitated by the addition of sodium nitrite.

$$[H_2ONCSCo(C_2H_8N_2)_2](NO_3)_2 + 2NaNO_2 \rightarrow [NO_2NCSCo(C_2H_8N_2)_2]NO_2 + 2NaNO_3 + H_2O$$

A. Werner and R. Schmidt, Ann., **386**, 162 (1912) *20*

$[CoNH_3NO_3en_2](NO_3)_2$

$Na_2S_2O_6$ **III-611**

Solid sodium dithionate is added to an aqueous solution of cis nitratoammonodiethylenediamino cobaltic nitrate and the insoluble precipitate filtered off and dried. It is cis nitratoamminodiethylenediamino cobaltic dithionate and crystallizes with one H_2O.

$$[NO_3NH_3Co(C_2H_8N_2)_2](NO_3)_2 + Na_2S_2O_6 \rightarrow [NO_3NH_3Co(C_2H_8N_2)_2]S_2O_6 + 2NaNO_3$$

A. Werner and C. Rix, Ann., **386**, 215 (1912) *20*

$[CoNH_3H_2Oen_2](NO_3)_3$

HNO_3 **III-612**

Cis aquoamminodiethylenediamino cobaltic nitrate is added to concentrated nitric acid and the solution evaporated to dryness on the water bath. The residue is dissolved in a small volume of water and allowed to stand. Cis nitratoamminodiethylenediamino cobaltic nitrate deposits as small orange red lustrous crystals which may be purified by recrystallization from hot water.

$$[H_2ONH_3Co(C_2H_8N_2)_2](NO_3)_3 \rightarrow [NO_3NH_3Co(C_2H_8N_2)_2](NO_3)_2 + H_2O$$

A. Werner and C. Rix, Ann., **386**, 214 (1912) *20*

$[Co(NO_2)_2en_2](NO_3)_3$

HNO_3 **III-613**

Dinitritodiethylenediaminocobalt trinitrate is dissolved in nitric acid (sp. gr. 1.4) and heated on the water bath until no more nitric oxide is evolved and the solution becomes dark red in color. The solution is evaporated to dryness, dissolved in hot concentrated nitric acid and placed in a desiccator over potassium hydroxide. Dark red triclinic crystals of dinitratodiethylenediaminocobalt trinitrate form.

$$[Co(NO_2)_2(C_2H_8N_2)_2](NO_3)_3 + 2HNO_3 \rightarrow [Co(NO_3)_2(C_2H_8N_2)_2](NO_3)_3 + H_2O + NO_2 + NO$$

A. Werner and C. Kreutzer, Ann., **351**, 79 (1907) *20*

$[Co(NH_3)_2en_2](NO_3)_3$

KI **III-614**

7 Grams of diamminodiethylenediamincobalt trinitrate are dissolved in 25 cc of hot water and heated to 60°C. 20 Grams of potassium iodide are added and, after a short time, bright yellow crystals of diamminodiethylenediamino cobalt triiodide form.

$$[Co(NH_3)_2(C_2H_8N_2)_2](NO_3)_3 + 3KI \rightarrow [Co(NH_3)_2(C_2H_8N_2)_2]I_3 + 3KNO_3$$

A. Werner and C. Kreutzer, Ann., **351**, 81 (1907) *20*

$[CoBrNCSen_2]SCN$

HBr **III-615**

Trans bromoisothiocyanatodiethylenediamino cobaltic thiocyanate is triturated with concentrated hydrobromic acid. The solid dissolves and the solution soon sets to a solid mass of bluish red crystals which after 20 minutes are pressed on a porous plate. The process of trituration with hydrobromic acid is repeated and the residue washed free of acid with alcohol and dried by washing with ether. The precipitate is recrystallized from hot water and dark blue prisms of trans bromoisothiocyanatodiethylenediamino cobaltic bromide with two H_2O deposit.

$$[BrNCSCo(C_2H_8N_2)_2]SCN + HBr \rightarrow [BrNCSCo(C_2H_8N_2)_2]Br + HSCN$$

A. Werner and R. Schmidt, Ann., **386**, 146 (1912) *20*

$[CoClNCSen_2]SCN$

HBr **III-616**

Trans chloroisothiocyanatodiethylenediamino cobaltic thiocyanate is triturated with hydrobromic acid and the resulting precipitate pressed on a porous plate. This residue is dissolved in a small volume of water and concentrated hydrobromic acid added when small lustrous blue violet crystals of trans chloroisothiocyanatodiethylenediamino cobaltic bromide deposit. They are recrystallized from hot water and these crystals contain two H_2O. The compound dissolves easily in water to form a blue violet solution.

$$[ClNCSCo(C_2H_8N_2)_2]SCN + HBr \rightarrow [ClNCSCo(C_2H_8N_2)_2]Br + HSCN$$

A. Werner and R. Schmidt, Ann., **386**, 133 (1912) *20*

$[CoOHen_2]SCN$

HBr **III-617**

Trans hydroxoisothiocyanatodiethylenediamino cobaltic thiocyanate is added to a small volume of water and an excess of concentrated hydrobromic acid added. The solution is allowed to concentrate in a vacuum over sulphuric acid. Dark red crystals of trans aquoisothiocyanatodiethylenediamino cobaltic bromide with two H_2O deposit.

$$[OHNCSCo(C_2H_8N_2)_2]SCN + 2HBr \rightarrow [NCSH_2OCo(C_2H_8N_2)_2]Br_2 + HSCN$$

A. Werner and R. Schmidt, Ann., **386**, 160 (1912) *20*

$[CoOHNCSen_2]SCN$

HBr **III-618**

KSCN

Trans hydroxoisothiocyanatodiethylenediamino cobaltic thiocyanate is treated with concentrated hydrobromic acid and the acid removed by evaporation. The aqueous solution of the residue is then treated with solid potassium thiocyanate and dark green crystals of trans bromoisothiocyanatodiethylenediamino cobaltic thiocyanate deposit. The salt is soluble in concentrated hydrobromic acid to give a violet solution.

(a) $[OHNCSCo(C_2H_8N_2)_2]SCN + 2HBr \rightarrow [BrNCSCo(C_2H_8N_2)_2]Br + H_2O + HSCN$

(b) $[BrNCSCo(C_2H_8N_2)_2]Br + KSCN \rightarrow [BrNCSCo(C_2H_8N_2)_2]SCN + KBr$

A. Werner and R. Schmidt, Ann., **386**, 159 (1912) *20*

$[CoClNO_2en_2]SCN$

($HC_2H_3O_2$) **III-619**

20 Grams of cis chloronitritodiethylenediamino cobaltic thiocyanate are dissolved in water acidified with acetic acid by heating on the water bath. Heating is continued until the volume of the solution is reduced to one half the original. During this concentration the color changes from red to red brown then to brown and, upon cooling, brownish yellow crystals of cis nitritoisothiocyanatodiethylenediamino cobaltic chloride with one H_2O separate.

$$[ClNO_2Co(C_2H_8N_2)_2]SCN \rightarrow [NO_2NCSCo(C_2H_8N_2)_2]Cl$$

A. Werner and C. Rix, Ann., **386**, 231 (1912) *20*

$[CoNO_2NCSen_2]SCN$

HCl **III-620**

5 Grams of cis nitritoisothiocyanatodiethylenediamino cobaltic thiocyanate are dissolved in 100 cc of water slightly acidified with acetic acid by heating if necessary. 1.5 cc of concentrated hydrochloric acid when added dropwise, precipitation begins almost immediately. The solution is warmed on the water bath and gas is evolved. The color changes from brown to dark red. The solution is filtered and the filtrate concentrated on the water bath. It changes to a red violet color and a red precipitate forms. It is recrystallized from hot water acidified with acetic acid and red needles of cis diisothiocyanatodiethylenediamino cobaltic chloride with one half H_2O deposit.

$$2[NO_2NCSCo(C_2H_8N_2)_2]SCN + 4HCl \rightarrow [(NCS)_2Co(C_2H_8N_2)_2]Cl + [Cl_2Co(C_2H_8N_2)_2]Cl + 2HNO_2 + 2HSCN$$

A. Werner and C. Rix, Ann., **386**, 195 (1912) *20*

$[CoNO_2NCSen_2]SCN$

HCl **III-621**

2 Grams of cis nitroisothiocyanatodiethylenediamino cobaltic thiocyanate are dissolved in 40 cc of water acidified with acetic acid by heating on the water bath. Hydrochloric acid is added dropwise until crystallization begins. Gas is evolved from the decomposition of nitrous acid and the color of the solution changes from brown to dark red. Dark red crystals of cis diisothiocyanatodiethylenediamino cobaltic chloride with one half H_2O deposit. The salt may be recrystallized from hot water containing acetic acid.

$$[NO_2NCSCo(C_2H_8N_2)_2]SCN + HCl \rightarrow [(NCS)_2Co(C_2H_8N_2)_2]Cl + HNO_2$$

A. Werner and C. Rix, Ann., **386**, 238 (1912) *20*

$[CoNO_2NCSen_2]SCN$

HCl **III-622**

6 Grams of cis nitritoisothiocyanatodiethylenediamino cobaltic thiocyanate are dissolved in 20 cc of concentrated hydrochloric acid by heating. The solution is filtered and alcohol added to the filtrate. The precipitate which forms is recrystallized from a small volume of warm water and reddish brown tablet-like crystals of cis nitritoisothiocyanatodiethylenediamino cobaltic chloride with one H_2O deposit.

$$[NO_2NCSCo(C_2H_8N_2)_2]SCN + HCl \rightarrow [NO_2NCSCo(C_2H_8N_2)_2]Cl + HSCN$$

A. Werner and N. Gosling, Ann., **386**, 243 (1912) *20*

$[CoBrNCSen_2]SCN$

$HClO_4$ **III-623**

2 Grams of trans bromoisothiocyanatodiethylenediamino cobaltic thiocyanate are triturated twice with concentrated perchloric acid and allowed to stand for a few minutes. The residue is pressed on a porous plate and then washed with alcohol and ether. It is then recrystallized from warm water and dark blue fine needles of trans bromoisothiocyanatodiethylenediamino cobaltic perchlorate are deposited.

$$[BrNCSCo(C_2H_8N_2)_2]SCN + HClO_4 \rightarrow [BrNCSCo(C_2H_8N_2)_2]ClO_4 + HSCN$$

A. Werner and R. Schmidt, Ann., **386**, 148 (1912) *20*

$[CoOHNCSen_2]SCN$

HNO_3 **III-624**

NH_4NO_3

Trans hydroxoisothiocyanatodiethylenediamino cobaltic thiocyanate is dissolved in dilute nitric acid, the solution filtered, solid ammonium nitrate added and the mixture triturated. The resulting mass is dissolved in water and several volumes of alcohol added. Blue red needles of trans aquoisothiocyanatodiethylenediamino cobaltic nitrate with one H_2O deposit.

$$[NCSOHCo(C_2H_8N_2)_2]SCN + HNO_3 + NH_4NO_3 \rightarrow [NCSH_2OCo(C_2H_8N_2)_2](NO_3)_2 + NH_4SCN$$

A. Werner and R. Schmidt, Ann., **386**, 161 (1912) *20*

$[CoClNCSen_2]SCN$

H_2O_2 **III-625**

HCl

$BaCl_2$

4 Grams of trans chloroisothiocyanatodiethylenediamino cobaltic thiocyanate are covered with 6 cc of water and 2 cc of dilute sulfuric acid (1:3) added with cooling. 10 cc of concentrated hydrogen peroxide are added, the solution stirred and allowed to stand 2 hours. Concentrated hydrochloric acid is added and the solution concentrated on the water bath. Upon cooling, bright red leaflets of trans chloroamminodiethylenediamino cobaltic chloride-sulfate precipitate. These are recrystallized three times from concentrated hydrochloric acid. Thick red crystals are obtained which still give a strong reaction for sulfates. These crystals are dissolved in water and a dilute solution of barium chloride added as long as a precipitate forms. The solution is filtered to remove barium sulfate and concentrated on the water bath. Alcohol is added to the filtrate and bright red prisms of trans chloroamminodiethylenediamino cobaltic chloride with one H_2O deposit. The salt is very soluble in water.

(a) $2[ClSCNCo(C_2H_8N_2)_2]SCN + 8H_2O_2 + 2HCl + H_2SO_4$
$\rightarrow [ClNH_3Co(C_2H_8N_2)_2]_2Cl_2 \cdot SO_4 + 2H_2SO_4 + 2CO_2 + 4H_2O + 2HSCN$

(b) $[ClNH_3Co(C_2H_8N_2)_2]_2Cl_2 \cdot SO_4 + BaCl_2$
$\rightarrow 2[ClNH_3Co(C_2H_8N_2)_2]Cl_2 + \underline{BaSO_4}$

A. Werner and R. Schmidt, Ann., **386**, 170 (1912) *20*

$[CoClNCSen_2]SCN$

KOH **III-626**

Trans chloroisothiocyanatodiethylenediamino cobaltic thiocyanate is treated with a concentrated aqueous solution of potassium hydroxide (1:4) and the mixture warmed slightly. The salt dissolves to form an intensive red colored solution and brownish red leafy crystals separate. Cooling the solution increases the yield. These crystals are removed, washed thoroughly with alcohol and ether then recrystallized from dilute potassium hydroxide solution. The compound formed is hydroxoisothiocyanatodiethylenediamino cobaltic thiocyanate (trans form). The crystals have one H_2O and dissolve in water to form alkaline solutions.

$[NCSClCo(C_2H_8N_2)_2]SCN + KOH$
$\rightarrow [OHNCSCo(C_2H_8N_2)_2]SCN + KCl$

A. Werner and R. Schmidt, Ann., **386**, 158 (1912) *20*

$[CoClNCSen_2]SCN$

NH_3

KSCN

III-627

Dry trans chloroisothiocyanatodiethylenediamino cobaltic thiocyanate is treated with sufficient liquid ammonia to form a solution. The ammonia is allowed to evaporate and an orange red mass remains which is taken up in warm water acidified with acetic acid. As this solution cools, the difficultly soluble trans isothiocyanatoamminodiethylenediamino cobaltic thiocyanate deposits. This compound is removed and solid potassium thiocyanate added to the filtrate when cis isothiocyanatoamminodiethylenediamino cobaltic thiocyanate deposits.

$$[ClNCSCo(C_2H_8N_2)_2]SCN + NH_3 + KSCN \rightarrow [NCSNH_3Co(C_2H_8N_2)_2](SCN)_2 + KCl$$

A. Werner and C. Rix, Ann., **386**, 206 (1912) *20*

$[CoNH_3NCSen_2](SCN)_2$

HBr

$Na_2S_2O_6$

III-628

Trans isothiocyanatoamminodiethylenediamino cobaltic thiocyanate is added to concentrated hydrobromic acid and triturated. The residue is pressed on a porous plate, washed with alcohol and dissolved in a small volume of water. Solid sodium dithionate is added and the precipitate which forms is recrystallized from hot water. Trans isothiocyanatoamminodiethylenediamino cobaltic bromide dithionate deposits as brownish red prisms with two H_2O.

$$2[NCSNH_3Co(C_2H_8N_2)_2](SCN)_2 + 2HBr + Na_2S_2O_6 \rightarrow [NCSNH_3Co(C_2H_8N_2)_2]_2Br_2 \cdot S_2O_6 + 2NaSCN + 2HSCN$$

A. Werner and C. Rix, Ann., **386**, 210 (1912) *20*

$[CoClNCSen_2](SCN)_2$

NH_3 **III-629**

KSCN

Trans chloroisothiocyanatodiethylenediamino cobaltic thiocyanate is treated with liquid ammonia and the ammonia allowed to evaporate. The residue consists of both the trans and cis isomers of isothiocyanatoamminodiethylenediamino cobaltic thiocyanate. The trans compound is deposited from the solution of the mixture in warm water slightly acidified with acetic acid but considerable potassium thiocyanate must be added to the filtrate from the solution, after removal of the trans salt, to precipitate the cis salt. The cis form is recrystallized from hot water and is obtained as red orange needles.

$$[ClNCSCo(C_2H_8N_2)_2]SCN + NH_3 + KSCN \rightarrow [NCSNH_3Co(C_2H_8N_2)_2](SCN)_2 + KCl$$

A. Werner and C. Rix, Ann., **386**, 210 (1912) *20*

$[CoNH_3NCSen_2](SCN)_2$

NH_4I **III-630**

Trans isothiocyanatoamminodiethylenediamino cobaltic thiocyanate is added to a small volume of water and solid ammonium iodide added. The mixture is triturated and the residue sucked dry. It is recrystallized from hot concentrated aqueous solution and small cherry red crystals of trans isothiocyanatoamminodiethylenediamino cobaltic iodide with one H_2O are obtained.

$$[NCSNH_3Co(C_2H_8N_2)_2](SCN)_2 + 2NH_4I \rightarrow [NCSNH_3Co(C_2H_8N_2)_2]I_2 + 2NH_4SCN$$

A. Werner and C. Rix, Ann., **386**, 210 (1912) *20*

$[CoNH_3NCSen_2](SCN)_2$

$Na_2S_2O_6$ **III-631**

Solid sodium dithionate is added to a solution of cis isothiocyanatoamminodiethylenediamino cobaltic thiocyanate and the difficultly soluble cis isothiocyanatoamminodiethylenediamino cobaltic dithionate precipitates. It is recrystallized from hot water and obtained as orange red leaflets.

$$[NCSNH_3Co(C_2H_8N_2)_2](SCN)_2 + Na_2S_2O_6 \rightarrow [NCSNH_3Co(C_2H_8N_2)_2]S_2O_6 + 2NaSCN$$

A. Werner and C. Rix, Ann., **386**, 207 (1912) *20*

$[CoClH_2Oen_2]SO_4$

HBr **III-632**

Cis chloroaquodiethylenediamino cobaltic sulfate is dissolved in ammonium hydroxide and concentrated hydrobromic acid added. Cis chlorobromodiethylenediamino cobaltic bromide is precipitated.

$$[Cl(H_2O)Co(C_2H_8N_2)_2]SO_4 + 2HBr \rightarrow [Cl(H_2O)Co(C_2H_8N_2)_2]Br_2 + H_2SO_4$$

A. Werner, L. Gerb, S. Lorie and J. Rapiport, Ann., **386**, 123 (1912) *20*

$[CoClH_2Oen_2]SO_4$

HCl **III-633**

25 Grams of cis chloroaquodiethylenediamino cobaltic sulfate are treated with 30 cc of concentrated hydrochloric acid. The sulfate dissolves to form a dark red solution from which red violet crystals of cis chloroaquodiethylenediamino cobaltic chloride deposits. The salt is easily soluble in water to form a reddish violet colored solution.

$$[Cl(H_2O)Co(C_2H_8N_2)_2]SO_4 + 2HCl \rightarrow [Cl(H_2O)Co(C_2H_8N_2)_2]Cl_2 + H_2SO_4$$

A. Werner, L. Gerb, S. Lorie and J. Rapiport, Ann., **386**, 123 (1912) *20*

$[Co(NH_3)_3Cl(H_2O)_2]SO_4$

KBr **III-634**

Hexamminotrioldicobalt sulfate is prepared by leaving chlorodiaquo-triammino-cobalt sulfate with potassium bromide for several days at ordinary temperature.

$$4[Cl(H_2O)_2(NH_3)_3Co]SO_4 + 6KBr \rightarrow K_2SO_4 + 5H_2O + 4KCl + [(NH_3)_3Co(OH)_3 \cdot Co(NH_3)_3)]_2(SO_4)_3 + (3HBr + 1.5\ Br_2)$$

A. Werner, Ber., **40**, 4605 (1907)
Ref., A. McKenzie, J. Chem. Soc. (London), **94:2**, 42 (1908) *57*

$[Co(NH_3)_3Cl(H_2O)_2]SO_4$

NaOH **III-635**

Chlorodiaquotriamminocobaltic sulfate reacts with sodium hydroxide solution yielding trihydroxodicobalthexammino sulfate.

$$4[Co(H_2O)_2(NH_3)_3Cl]SO_4 + 6NaOH \rightarrow [Co_2(OH)_3(NH_3)_6]_2(SO_4)_3 + Na_2SO_4 + 4NaCl + (8H_2O)$$

A. Werner, Ber., **40**, 4834 (1907)
Ref., J. J. Sudborough, J. Chem. Soc. (London), **94:2**, 43 (1908) *57*

$[CoNH_3NCSen_2]S_2O_6$

$AgNO_3$ **III-636**

1.1 Grams of cis isothiocyanatoamminodiethylenediamino cobaltic dithionate are added to 90 cc of water 1.5 to 2 equivalents of silver nitrate in aqueous solution added and the solution warmed until solution is complete. The solution is filtered quickly and the filtrate allowed to stand. Small lustrous yellow crystals deposit. These are washed with alcohol and ether. The salt is cis silver isothiocyanato-amminodiethylenediamino cobaltic nitro dithionate.

$$[NCSNH_3Co(C_2H_8N_2)_2]S_2O_6 + AgNO_3 \rightarrow [AgSCNNH_3Co(C_2H_8N_2)_2]NO_3 \cdot S_2O_6$$

A. Werner and C. Rix, Ann., **386**, 209 (1912) *20*

$[CoNH_3SCNen_2]S_2O_6$

$AgNO_3$ **III-637**

When silver nitrate solution is added to isothiocyanatoammino-diethylenediaminocobaltic dithionate, golden yellow crystals of an addition compound are precipitated.

$$[SCNCo(NH_2C_2H_4NH_2)_2NH_3]S_2O_6 + AgNO_3 \rightarrow \underline{[SCNCo(NH_2C_2H_4NH_2)_2NH_3]S_2O_6 \cdot AgNO_3}$$

T. S. Price and S. A. Brazier, J. Chem. Soc. (London), **107**, 1715 (1915) *57*

$[CoH_2ONCSen_2]S_2O_6$

$AgNO_3$ **III-638**

$HClO_4$

Cis aquoisothiocyanatodiethylenediamino cobaltic dithionate is dissolved in a small volume of water slightly acidified with acetic acid and a concentrated aqueous solution of silver nitrate added. The color of the solution changes from red orange to yellowish and, upon addition of perchloric acid, an orange precipitate forms. This is removed, washed with alcohol, dissolved in a small volume of water and reprecipitated by the addition of perchloric acid. Orange crystals of cis aquosilverthiocyanatodiethylenediamino cobaltic perchlorate with two H_2O deposit.

$$[H_2ONCSCo(C_2H_8N_2)_2]S_2O_6 + AgNO_3 + 2HClO_4 \rightarrow [H_2OAgNCSCo(C_2H_8N_2)_2]NO_3(ClO_4)_2 + H_2S_2O_6$$

A. Werner and R. Schmidt, Ann., **386**, 157 (1912) *20*

$[CoNH_3NO_2en_2]S_2O_6$

HCl

III-639

Trans nitritoamminodiethylenediamino cobaltic dithionate is evaporated to dryness several times with concentrated hydrochloric acid. The original yellow colored solution changes to red as the reaction progresses. As the solution cools, ruby red plates of trans nitritoamminodiethylenediamino cobaltic chlorobisulfate deposit. Aqueous solutions of the salt have a strong acid reaction.

$$[NO_2NH_3Co(C_2H_8N_2)_2]S_2O_6 + HCl \rightarrow [NO_2NH_3Co(C_2H_8N_2)_2]ClHSO_4 + SO_2$$

A. Werner and R. Schmidt, Ann., **386**, 172 (1912) *20*

$[CoH_2ONCSen_2]_2S_2O_6$

HCl

HClO$_4$

LiNO$_3$

III-640

1.5 Grams of cis aquoisothiocyanatodiethylenediamino cobaltic dithionate are added to 5 cc of concentrated hydrochloric acid and the solution warmed until the liquid has assumed a violet red color. Absolute alcohol is added and the violet red precipitate is pressed on a porous plate. This residue is rubbed with perchloric acid, absolute alcohol added and the precipitate of the perchlorate dissolved in a small volume of water, then lithium nitrate added. Cis chloroisothiocyanatodiethylenediamino cobaltic nitrate precipitates as a dark red powder which is recrystallized from hot water and dark red crystals of the compound are obtained.

(a) $$[NCSH_2OCo(C_2H_8N_2)_2]_2S_2O_6 + 4HCl \rightarrow 2[NCSClCo(C_2H_8N_2)_2]Cl + H_2S_2O_6 + 2H_2O$$

(b) $$[SCNClCo(C_2H_8N_2)_2]Cl + HClO_4 \rightarrow [SCNClCo(C_2H_8N_2)_2]ClO_4 + HCl$$

(c) $$[SCNClCo(C_2H_8N_2)_2]ClO_4 + LiNO_3 \rightarrow [SCNClCo(C_2H_8N_2)_2]NO_3 + LiClO_4$$

A. Werner and R. Schmidt, Ann., **386**, 156 (1912) *20*

$[CoH_2ONCSen_2]S_2O_6$

KSCN **III-641**

Cis isothiocyanatoaquodiethylenediamino cobaltic dithionate in concentrated aqueous solution is treated with potassium thiocyanate and the solution heated then allowed to stand. The color of the solution changes to red and red crystals of cis isothiocyanatodiethylenediamino cobaltic dithionate deposit.

$$2[NCSH_2OCo(C_2H_8N_2)_2]S_2O_6 + 2KSCN \rightarrow [(NCS)_2Co(C_2H_8N_2)_2]_2S_2O_6 + 2H_2O + K_2S_2O_6$$

A. Werner and R. Schmidt, Ann., **386**, 157 (1912) *20*

$[CoNH_3Bren_2]S_2O_6$

NH_4I **III-642**

11.5 Grams of trans bromoamminodiethylenediamino cobaltic dithionate are mixed with 23 cc of cold water and added to a solution of 60 grams of ammonium iodide in 42 cc of water. The dithionate dissolves and a brown precipitate forms. This precipitate is removed, dissolved in 250 cc of cold water and 60 grams of ammonium iodide added. Reddish brown lustrous needles of trans bromoamminodiethylenediamino cobaltic iodide with one H_2O deposit.

$$[BrNH_3Co(C_2H_8N_2)_2]S_2O_6 + 2NH_4I \rightarrow [BrNH_3Co(C_2H_8N_2)_2]I_2 + (NH_4)_2S_2O_6$$

A. Werner and W. Boes, Ann., **386**, 183 (1912) *20*

$[CoNH_3NCSen_2]S_2O_6$

NH_4I **III-643**

Cis isothiocyanatoamminodiethylenediamino cobaltic dithionate is suspended in water, solid ammonium iodide added and the mixture triturated. The salt dissolves and in a short time an intense red colored precipitate forms. This is removed by filtration, the residue sucked dry as possible and then recrystallized from a small volume of water by the addition of ammonium iodide. Short reddish brown, prismatic crystals of cis isothiocyanatoamminodiethylenediamino cobaltic iodide deposit.

$$[NCSNH_3Co(C_2H_8N_2)_2]S_2O_6 + 2NH_4I \rightarrow [NCSNH_3Co(C_2H_8N_2)_2]I_2 + (NH_4)_2S_2O_6$$

A. Werner and C. Rix, Ann., **386**, 207 (1912) *20*

$[CoH_2ONCSen_2]S_2O_6$

$NaNO_2$ **III-644**

2 Grams of cis aquoisothiocyanatodiethylenediamino cobaltic dithionate are dissolved in a small volume of water slightly acidified with acetic acid, 0.5 gram of sodium nitrite added and the solution warmed until it becomes yellow. The solution is cooled and fine yellow needles of cis nitritoisothiocyanatodiethylenediamino cobaltic dithionate deposit. The salt may be recrystallized from hot water.

$$2[H_2ONCSCo(C_2H_8N_2)_2]S_2O_6 + 2NaNO_2 \rightarrow [NO_2NCSCo(C_2H_8N_2)_2]_2S_2O_6 + 2H_2O + Na_2S_2O_6$$

A. Werner and R. Schmidt, Ann., **386**, 157 (1912) *20*

$[CoH_2OOHen_2]S_2O_6$

Δ **III-645**

Tetraethylenediamine-mu-diol dicobaltic dithionate is formed when hydroxoaquodiethylenediaminocobaltic dithionate is heated to constant weight at 100° to 110°C.

$$2[H_2OOHCo(C_2H_8N_2)_2]S_2O_6 \rightarrow [(C_2H_8N_2)_2CoOHOHCo(C_2H_8N_2)_2](S_2O_6)_2 + 2H_2O$$

A. Werner, Ann., **375**, 84 (1910) *20*

$[CoNH_3H_2Oen_2]X_3$

HNO_2 **III-646**

Cis aquoamminodiethylenediamino cobaltic salts react with nitrous acid to form cis nitritoamminodiethylenediamino cobaltic salts.

$$[H_2ONH_3Co(C_2H_8N_2)_2]X_3 + HNO_2 \rightarrow [NO_2NH_3Co(C_2H_8N_2)_2]X_2 + HX + H_2O$$

A. Werner and W. Boes, Ann., **386**, 217 (1912) *20*

$[CoOHBr_2en_2]_2$

HBr **III-647**

5 Grams of tetraethylenediamine dicobaltic hydroxy dibromide is added to 10 cc of concentrated hydrobromic acid at −12°C and the reaction product pressed on a porous plate. This residue is washed with absolute alcohol. A mixture of cis diaquodiethylenediamine cobaltic bromide and cis dibromodiethylenediamine cobaltic bromide is formed. These are separated by fractional extraction. The diaquo salt is soluble in water while the dibromo salt is much less soluble.

$$[CoOHBr_2(C_2H_8N_2)_2]_2 + 2HBr \rightarrow [Br_2Co(C_2H_8N_2)_2]Br + [(H_2O)_2Co(C_2H_8N_2)_2]Br_3$$

A. Werner, L. Gerb, S. Lorie and J. Rapiport, Ann., **386**, 113 (1912) *20*

$[Co_2(NH_3)_6Br_4NH_2]Br$

HNO_3 **III-648**

Tetrabromohexamminomuaminodicobalt chloride is triturated with concentrated nitric acid and quickly filtered. This operation is repeated two or three times. The compound formed is tetrabromohexamminomuaminodicobaltic nitrate which has two H_2O.

$$[(NH_3)_3Br_2CoNH_2CoBr_2(NH_3)_3]Br + HNO_3 \rightarrow [(NH_3)_3Br_2CoNH_2CoBr_2(NH_3)_3]NO_3 + HBr$$

A. Werner and S. Malmgren, Ann., **375**, 98 (1910) *20*

$[Co_2(NH_3)_6Br_2O_2NH_2]Br_2$

NH_3 **III-649**

Unsymmetrical dibromohexamminomuaminoperoxodicobaltic bromide when treated with liquid ammonia is converted quantitatively into octamminomuaminoperoxodicobaltic bromide.

$$[(NH_3)_2Br_2CoNH_2O_2Co(NH_3)_4]Br_2 + 2NH_3 \rightarrow [(NH_3)_4CoNH_2O_2Co(NH_3)_4]Br_4$$

A. Werner and M. Grigorieff, Ann., **375**, 114 (1910) *20*

$[Co_2(NH_3)_6Br_2O_2NH_2]Br_2$

NH_3 **III-650**

H_2SO_4

Symmetrical dibromohexamminomuaminoperoxodicobaltic bromide is treated with sufficient liquid ammonia to completely dissolve the bromide. The ammonia is allowed to evaporate from the greenish brown solution, the residue dissolved in water and dilute sulfuric acid added. Pale green, silvery lustrous crystals of octamminomuaminoperoxodicobaltic sulfate precipitate. These are converted to the nitrate by triturating with nitric acid.

(a) $[(NH_3)_3BrCoNH_2O_2CoBr(NH_3)_3]Br_2 + 2NH_3 \rightarrow [(NH_3)_4CoNH_2O_2Co(NH_3)_4]Br_4$

(b) $[(NH_3)_4CoNH_2O_2Co(NH_3)_4]Br_4 + 2H_2SO_4 \rightarrow [(NH_3)_4CoNH_2O_2Co(NH_3)_4](SO_4)_2 + 4HBr$

A. Werner and M. Grigorieff, Ann., **375**, 109 (1910) *20*

$[Co_2(NH_3)_6Br_2OHNO_2]Br_2$

$NaNO_3$ **III-651**

0.5 Gram of dibromohexamminomuolnitrosodicobalic bromide is added to a saturated aqueous solution of sodium nitrate and solid sodium nitrate added while the solution is being triturated. After a few minutes the crystals are removed by filtration and the process of treatment with sodium nitrate repeated. Dibromohexamminomuolnitrosodicobaltic nitrate is formed.

$[(NH_3)_3BrCoOHNO_2CoBr(NH_3)_3]Br_2 + 2NaNO_3 \rightarrow [(NH_3)_3BrCoOHNO_2CoBr(NH_3)_3](NO_3)_2 + 2NaBr$

A. Werner and E. Welti, Ann., **375**, 129 (1910) *20*

$[Co_2(NH_3)_6Br_2OHNO_2]Br_2$

$Na_2S_2O_6$ **III-652**

0.5 Gram of dibromohexamminomuolnitrosodicobalitc bromide is added to a saturated aqueous solution of sodium dithionate and solid sodium dithionate added while the mixture is being triturated. The crystals are removed by filtration and the crystals again treated with sodium dithionate. The crystals are dibromohexamminomuolnitrosodicobaltic dithionate. The compound is only slightly soluble in water.

$[(NH_3)_3BrCoOHNO_2CoBr(NH_3)_3]Br_2 + Na_2S_2O_6 \rightarrow [(NH_3)_3BrCoOHNO_2CoBr(NH_3)_3]S_2O_6 + 2NaBr$

A. Werner and E. Welti, Ann., **375**, 129 (1910) *20*

$[Co_2(NH_3)_6O_2NH_2OH]Br_3$

AgCl **III-653**

A concentrated aqueous solution of hexamminomuolaminoperoxodicobaltic bromide is shaken with freshly precipitated silver chloride for 2–2½ hours, filtered and ammonium chloride or alcohol added to the filtrate. Crystals of hexamminomuolaminoperoxodicobaltic chloride with one H_2O precipitate. The compound is crystallized from a small volume of water.

$$[(NH_3)_3CoNH_2OHO_2Co(NH_3)_3]Br_3 + 3AgCl \rightarrow [(NH_3)_3CoNH_2OHO_2Co(NH_3)_3]Cl_3 + 3AgBr$$

A. Werner and M. Grigorieff, Ann., **375**, 106 (1910) *20*

$[Co_2(NH_3)_6O_2NH_2OH]Br_3$

$AgNO_3$ **III-654**

Hexamminomuolaminoperoxodicobaltic bromide is dissolved in water, the solution cooled and the calculated amount of silver nitrate in solution added. The solution is filtered and alcohol added to the filtrate when dark green crystals of hexamminomuolaminoperoxodicobaltic nitrate with one H_2O deposit.

$$[(NH_3)_3CoNH_2OHO_2Co(NH_3)_3]Br_3 + 3AgNO_3 \rightarrow [(NH_3)_3CoNH_2OHO_2Co(NH_3)_3](NO_3)_3 + 3AgBr$$

A. Werner and A. Grigorieff, Ann., **375**, 107 (1910) *20*

$[Co_2(NH_3)_6(OH)_3]Br_3$

$C_2H_4O_2$ **III-655**

A solution of hexamminomutrioldicobaltic bromide is treated with acetic acid and heated on the water bath. Large violet red prisms of hexamminomuacetatodioldicobaltic bromide with one H_2O deposit. The water of crystallization is lost at 110°C. The salt dissolves in water and the solution is a bright rose color.

$$[(NH_3)_3Co(OH)_3Co(NH_3)_3]Br_3 + HC_2H_3O_2 \rightarrow [(NH_3)_3Co(OH)_2(C_2H_3O_2)Co(NH_3)_3]Br_3 + H_2O$$

A. Werner and M. Grigorieff, Ann., **375**, 115 (1910) *20*

$[Co_2(NH_3)_6OHNH_2OH]Br_3$

HBr **III-656**

An aqueous solution of hexamminomuaminodioldicobaltic bromide is warmed with concentrated hydrobromic acid on the water bath. Small brownish green crystals of tetrabromohexammonomuaminodicobaltic bromide with two H_2O deposit. The compound is insoluble in cold water and its water of crystallization is lost when heated to 70–80°C.

$$[(NH_3)_3CoNH_2OHOHCo(NH_3)_3]Br_3 + 2HBr \rightarrow [(NH_3)_3Br_2CoNH_2CoBr_2(NH_3)_3]Br + 2H_2O$$

A. Werner and S. Malmgren, Ann., **375**, 97 (1910) *20*

$[Co_2(NH_3)_6(OH)_2NO_2]Br_3$

HBr **III-657**

0.5 Gram of hexamminomudiolnitrosodicobaltic bromide is treated with concentrated hydrobromic acid and the solution warmed over a small flame. The orange color of the solution changes to gray and, upon cooling, faint green colored crystals of dibromohexamminomuolnitrosodicobaltic bromide deposit.

$$[(NH_3)_3CoOHOHNO_2Co(NH_3)_3]Br_3 + HBr \rightarrow [(NH_3)_3BrCoOHNO_2CoBr(NH_3)_3]Br_2 + H_2O$$

A. Werner and E. Welti, Ann., **375**, 128 (1910) *20*

$[Co_2(NH_3)_6O_2NH_2OH]Br_3$

HBr **III-658**

A concentrated aqueous solution of hexamminomuolaminoperoxodicobaltic bromide is treated with concentrated hydrobromic acid and warmed. Small, flat greenish crystals of symmetrical dibromohexamminomuaminoperoxodicobaltic bromide with two H_2O separate. The compound is only slightly soluble in water.

$$[(NH_3)_3CoNH_2OHO_2Co(NH_3)_3]Br_3 + HBr \rightarrow [(NH_3)_3BrCoNH_2O_2CoBr(NH_3)_3]Br_2 + H_2O$$

A. Werner and M. Grigorieff, Ann., **375**, 108 (1910) *20*

$[Co_2(NH_3)_6O_2NH_2OH]Br_3$

HBr **III-659**

SO_2

Hexamminomuaminoperoxooldicobaltic bromide is covered with a saturated water solution of sulfur dioxide and warmed carefully until the solution turns dark. The reaction mixture is then gently heated with concentrated hydrobromic acid whereupon dihydrated hexamminomuaminotetrabromodicobaltic bromide precipitates.
The bromide may be converted to nitrate by the use of silver nitrate in dilute nitric acid solution.

$$2[(NH_3)_3CoNH_2O_2(OH)Co(NH_3)_3]Br_3 + 3SO_2 + 4HBr \rightarrow 2[(NH_3)_3CoNH_2Br_4Co(NH_3)_3]Br + 3H_2SO_4$$

A. Werner, Ann., **375**, 110 (1910) *41*

$[Co_2(NH_3)_6(OH)_2C_2H_3O_2]Br_3$

$HC_2H_3O_2$ **III-660**

A concentrated aqueous solution of hexamminomudiolacetatodicobaltic bromide is added to an equal volume of acetic acid and the solution concentrated on the water bath. As the solution cools, crystals of acetatoaquohexamminomuolacetatodicobaltic bromide deposit.

$$[(NH_3)_3CoOHOH(C_2H_3O_2)Co(NH_3)_3]Br_3 + HC_2H_3O_2 \rightarrow [(NH_3)_3H_2OCoOH(C_2H_3O_2)Co(C_2H_3O_2)(NH_3)_3]Br_3$$

A. Werner and M. Grigorieff, Ann., **375**, 120 (1910) *20*

$[Co_2(NH_3)_6(OH)_3]Br_3$

$HC_2H_3O_2$ **III-661**

A saturated aqueous solution of hexamminomutrioldicobaltic bromide is added to one half its volume of acetic acid and the solution concentrated on the water bath to one half its volume. As the solution cools, red needles of acetatoaquohexamminomuolacetatodicobaltic bromide are formed. They are washed with alcohol and ether then dried over sulfuric acid.

$$[(NH_3)_3Co(OH)_3Co(NH_3)_3]Br_3 + 2HC_2H_3O_2 \rightarrow [(NH_3)_3H_2OCo(OH)(C_2H_3O_2)Co(C_2H_3O_2)(NH_3)_3]Br_3 + H_2O$$

A. Werner and M. Grigorieff, Ann., **375**, 119 (1910) *20*

$[Co_2(NH_3)_6(OH)_3]Br_3$

$HC_2H_3O_2$ **III-662**

$NaNO_2$

A warm aqueous solution of hexamminomutrioldicobaltic bromide is acidified with acetic acid and an excess of sodium nitrite added. The solution is stirred and in about ten minutes the color of the solution changes to orange red. Hydrobromic acid is added dropwise until no more gas is evolved. Intense orange colored crystals of hexamminomudiolnitritodicobaltic bromide with one H_2O deposit.

$$[(NH_3)_3Co(OH)_3Co(NH_3)_3]Br_3 + HC_2H_3O_2 + NaNO_2 \rightarrow [(NH_3)_3Co(OH)_2NO_2Co(NH_3)_3]Br_3 + C_2H_3O_2Na + H_2O$$

A. Werner, A. Grün and E. Bindschedler, Ann., **375**, 122 (1910) *20*

$[Co_2(NH_3)_6(OH)_2C_2H_3O_2]Br_3$

HCl **III-663**

Hexamminomuacetatodioldicobaltic bromide is treated with hydrochloric acid saturated with hydrochloric acid gas at 0°C. The crystals dissolve to a blue colored solution and soon crystals of cobaltictriammino chloride deposit.

$$[(NH_3)_3Co(C_2H_3O_2)(OH)_2Co(NH_3)_3]Br_3 + 6HCl \rightarrow 2[(NH_3)_3CoCl_3] + 2H_2O + HC_2H_3O_2 + 3HBr$$

A. Werner and M. Grigorieff, Ann., **375**, 116 (1910) *20*

$[Co_2(NH_3)_6(OH)_2C_2H_3O_2]Br_3$

KI **III-664**

A solution of hexamminomuacetatodioldicobaltic bromide is treated with solid potassium iodide or better with a saturated solution added dropwise. Red plates of hexamminomuacetatodioldicobaltic iodide with one H_2O deposit. These are washed with water and dried on a porous plate.

$$[(NH_3)_3Co(OH)_2(C_2H_3O_2)Co(NH_3)_3]Br_3 + 3KI \rightarrow [(NH_3)_3Co(OH)_2(C_2H_3O_2)Co(NH_3)_3]I_3 + 3KBr$$

A. Werner and M. Grigorieff, Ann., **375**, 117 (1910) *20*

$[Co_2(NH_3)_6(OH)_2C_2H_3O_2]Br_3$

KSCN **III-665**

Solid potassium thiocyanate is added to an aqueous solution of hexamminomuacetatodioldicobaltic bromide until it is saturated. A fine red crystalline precipitate of hexamminomuacetatodioldicobaltic thiocyanate forms. The salt has one H_2O.

$$[(NH_3)_3Co(OH)_2(C_2H_3O_2)Co(NH_3)_3]Br_3 + 3KSCN \rightarrow [(NH_3)_3Co(OH)_2(C_2H_3O_2)Co(NH_3)_3](SCN)_3 + 3KBr$$

A. Werner and M. Grigorieff, Ann., **375**, 117 (1910) *20*

$[Co_2(OH)_2en_4]Br_4$

HBr **III-666**

5 Grams of tetraethylenediaminomudioldicobaltic bromide are added to 10 cc of hydrobromic acid at 12°C and triturated. The residue is washed with alcohol until free from acid. The residue is cis-dibromodiethylenediaminocobaltic bromide.

$$[(C_2H_8N_2)_2Co(OH)_2Co(C_2H_8N_2)_2]Br_4 + 2HBr \rightarrow 2[Br_2Co(C_2H_8N_2)_2]Br + 2H_2O$$

A. Werner, Ann., **375**, 85 (1910) *41*

$[Co_2(NH_3)_8H_2OBrNH_2]Br_4$

H_2SO_4 **III-667**

Hydrated bromoaquooctaminomuaminodicobaltic bromide was washed with ice cold water and allowed to run into diluted (1:1) sulfuric acid. Dark brown crystals at once separated which gave the analysis for the corresponding sulfate.

$$[(NH_3)_4CoNH_2BrCo(H_2O)(NH_3)_4]Br_4 + H_2SO_4 \rightarrow [(NH_3)_4CoNH_2BrCo(NH_3)_4](SO_4)_2 + 4HBr + H_2O$$

A. Werner, Ann., **375**, 51 (1910) *41*

$[Co_2(NH_3)_8H_2OBrNH_2]Br_4$

H_2SO_4 **III-668**

Bromoaquooctamminomuaminodicobaltic bromide is added to ice cold water then treated with a solution of dilute sulfuric acid (1:1). Dark brown crystals of bromoaquooctamminomuaminodicobaltic sulfate deposit. The compound is almost completely insoluble in water.

$$[(NH_3)_4BrCoNH_2CoH_2O(NH_3)_4]Br_4 + 2H_2SO_4 \rightarrow [(NH_3)_4BrCoNH_2CoH_2O(NH_3)_4](SO_4)_2 + 4HBr$$

A. Werner, Ann., **375**, 51 (1910) *20*

$[Co_2(NH_3)_8NH_2OH]Br_4$

H_2SO_4 **III-669**

Octamminomuaminooldicobaltic bromide is boiled with 1:3 sulfuric acid until a violet solution is formed. Addition of concentrated hydrobromic acid to the cooled solution causes deposition of reddish-violet needles which can be recrystallized from fairly strong hydrobromic acid.

$$[(NH_3)_4CoNH_2OHCo(NH_3)_4]Br_4 + H_2SO_4 \rightarrow HBr + H_2O + [(NH_3)_4CoNH_2SO_4Co(NH_3)_4]Br_3$$

A. Werner, Ann., **375**, 53 (1910) *41*

$[Co_2(OH)_2en_4]Br_4$

KSCN **III-670**

A solution of tetraethylenediaminomudioldicobaltic bromide is treated with solid potassium thiocyanate and purple crystals of tetraethylenediaminomudioldicobaltic thiocyanate form. They have three H_2O.

$$[(C_2H_8N_2)_2CoOHOHCo(C_2H_8N_2)_2]Br_4 + 4KSCN \rightarrow [(C_2H_8N_2)_2CoOHOHCo(C_2H_8N_2)_2](SCN)_4 + 4KBr$$

A. Werner, Ann., **375**, 86 (1910) *20*

$[Co_2O_2NH_2en_4]Br_4$

NaI **III-671**

4 Grams of tetraethylenediaminomuaminoperoxodicobaltic bromide are dissolved in 15 cc of water, and 8 cc of ammonium hydroxide added then 10 grams of sodium iodide. A thick mass of dark brown crystals form. 1.5 Grams of sodium thiosulfate are added and the mass triturated. The residue is washed with alcohol, dissolved in 40 cc of water, filtered and small brown needles of tetraethylenediaminomuiminoperoxo dicobaltic iodide are precipitated by the addition of 40 grams of potassium iodide. The crystals have five H_2O.

$$[(C_2H_8N_2)_2CoNH_2O_2Co(C_2H_8N_2)_2]Br_4 + 3NaI \rightarrow [(C_2H_8N_2)_2CoNHO_2Co(C_2H_8N_2)_2]I_3 + 3NaBr + HBr$$

A. Werner, Ann., **375**, 74 (1910) *20*

$[Co_2(OH)_2en_4]Br_4$

$NaNO_3$ **III-672**

A saturated aqueous solution of tetraethylenediaminomudioldicobaltic bromide is treated with solid sodium nitrate and a purple mass of crystals precipitates. This is recrystallized from water by the addition of sodium nitrate and purple crystals of tetraethylenediaminomudioldicobaltic nitrate are deposited. The compound dissolves in water to form a blue-red solution.

$$[(C_2H_8N_2)_2CoOHOHCo(C_2H_8N_2)_2]Br_4 + 4NaNO_3 \rightarrow [(C_2H_8N_2)_2CoOHOHCo(C_2H_8N_2)_2](NO_3)_4 + 4NaBr$$

A. Werner, Ann., **375**, 86 (1910) *20*

$[Co_2(NH_3)_8BrH_2ONH_2]Br_4$

Decomp. **III-673**

An aqueous solution of bromoaquooctamminomuaminodicobaltic bromide is reddish brown in color but, upon standing, changes to carmine red color and red prismatic crystals of octamminomuaminooldicobaltic bromide deposit.

$$[(NH_3)_4BrCoNH_2CoH_2O(NH_3)_4]Br_4 \rightarrow [(NH_3)_4CoNH_2OHCo(NH_3)_4]Br_4 + HBr$$

A. Werner, Ann., **375**, 50 (1910) *20*

$[Co_2(NH_3)_6Cl_3O_2OH]Cl$

NH_3

HBr **III-674**

Hexamminotrichloromuolperoxodicobaltic chloride is treated with liquid ammonia. The excess of the latter is allowed to evaporate and hydrobromic acid is then added to the residue. The yellow product is a mixture of hexamminocobaltic bromide and bromopentamminocobaltic bromide.

$$2[(NH_3)_3Co(OH)O_2Cl_3Co(NH_3)_3]Cl + 18HBr + 10NH_3 \rightarrow 2[(NH_3)_3Co(NH_3)_3]Br_3 + 2[(NH_3)_5BrCo]Br_3 + 8HCl + 2Br_2 + 6H_2O$$

A. Werner, Ann., **375**, 138 (1910) *41*

$[Co_2(NH_3)_6H_2OClNH_2Cl_2]Cl_2$

$AgNO_3$ **III-675**

20 Grams of trichloroaquohexamminomuaminodicobaltic chloride (melanochloride) are finely pulverized and added to a solution of 36 grams of silver nitrate in 60 cc of water slightly acidified with nitric acid. This solution is heated to 50°C and filtered. The residue containing silver chloride is extracted with 60 cc portions of water slightly acidified with nitric acid until the extract is colorless. Alcohol is added to the combined extracts until a permanent turbidity exists; then the solution is placed in an ice bath. After a few hours a red crystalline mass precipitates. This is recrystallized from five times its volume of warm water by the addition of concentrated nitric acid and cooling. These crystals are diaquohexamminomuaminooldicobaltic nitrate and have two H_2O.

$$[(NH_3)_3ClH_2OCoNH_2CoCl_2(NH_3)_3]Cl_2 + 5AgNO_3 + 2H_2O \rightarrow [(NH_3)_3H_2OCoNH_2OHCoH_2O(NH_3)_3](NO_3)_4 + 5AgCl + HNO_3$$

A. Werner and J. Furstenberg, Ann., **375**, 89 (1910) *20*

$[Co_2(NH_3)_{10}(NO_2)_2(NO_3)_2]Cl_2$

$AgNO_3$ **III-676**

Flavo cobaltic chloride reacts with silver nitrate to form xantho cobaltic nitrate and silver chloride.

$$[Co_2(NH_3)_{10}(NO_2)_2(NO_3)_2]Cl_2 + 2AgNO_3 \rightarrow [Co_2(NH_3)_{10}(NO_2)_2](NO_3)_4 + 2AgCl$$

W. Gibbs, Ber., **6**, 831 (1873) *11*

$[Co_2(NH_3)_{10}(NO_2)_4]Cl_2$

$AgNO_3$ **III-677**

When a nitric acid solution of silver nitrate is added to a solution of xantho cobaltic chloride, xantho cobaltic nitrate is formed.

$$[Co_2(NH_3)_{10}(NO_2)_4]Cl_2 + 4AgNO_3 \rightarrow [Co_2(NH_3)_{10}(NO_2)_2](NO_3)_4 + 2AgCl + 2AgNO_2$$

W. Gibbs, Ber., **3**, 43 (1870) *11*

$[Co_2(NH_3)_{10}(NO_2)_4]Cl_2$

$C_2O_4(NH_4)_2$ **III-678**

Xantho cobaltic chloride reacts with ammonium oxalate to form xantho cobaltic oxalate.

$$[Co_2(NH_3)_{10}(NO_2)_4]Cl_2 + 2C_2O_4(NH_4)_2 \rightarrow [Co_2(NH_3)_{10}(NO_2)_2](C_2O_4)_2 + 2NH_4NO_2 + 2NH_4Cl$$

W. Gibbs, Ber., **3**, 43 (1870) *11*

$[Co_2(NH_3)_8(NO_2)_4]Cl_2$

HCl **III-679**

When dinitritotetraammino cobaltic chloride is boiled with dilute hydrochloric acid purpureo cobaltic chloride, cobaltous chloride, ammonium chloride, water, nitrogen, and nitrogen dioxide are formed.

$$4[Co_2(NH_3)_8(NO_2)_4]Cl_2 + 16HCl \rightarrow 4CoCl_2 + 4NH_4Cl + 18H_2O + 8\tfrac{1}{2}N_2 + 7NO_2 + 2Co_2(NH_3)_{10}Cl_6$$

W. Gibbs, Ber., **6**, 832 (1873) *11*

$[Co_2(NH_3)_6ClNH_2NO_2Cl]Cl_2$

HNO_3 **III-680**

Dichlorohexamminomuaminonitritodicobaltic chloride is mixed and rubbed with concentrated nitric acid, pressed as dry as possible on a porous plate and subsequently washed free from acid with alcohol. The solid is dissolved in cold water and nitric acid added. Dichlorohexamminomuaminonitritodicobaltic nitrate crystallizes as reddish brown plates. The compound is soluble in water.

$$[(NH_3)_3ClCoNH_2NO_2CoCl(NH_3)_3]Cl_2 + 2HNO_3 \rightarrow [(NH_3)_3ClCoNH_2NO_2CoCl(NH_3)_3](NO_3)_2 + 2HCl$$

A. Werner and S. Malmgren, Ann., **375**, 99 (1910) *20*

$[Co_2(NH_3)_6H_2OCl_2NH_2Cl]Cl_2$

HNO_3 **III-681**

Trichloroaquohexamminomuaminodicobaltic chloride is triturated twice with concentrated nitric acid and then dissolved in water. Nitric acid is added and dark violet colored crystals of trichloronitratohexamminomuaminodicobaltic nitrate deposit. The compound is soluble in water.

$$[(NH_3)_3Cl_2CoNH_2CoClH_2O(NH_3)_3]Cl_2 + 2HNO_3 \rightarrow [(NH_3)_3Cl_2CoNH_2CoClNO_2(NH_3)_3]NO_3 + H_2O + 2HCl$$

A. Werner and S. Malmgren, Ann., **375**, 97 (1910) *20*

$[Co_2(NH_3)_6OHClNO_2Cl]Cl_2$

HNO_3 **III-682**

3 Grams of dichlorohexamminomuolnitritodicobaltic chloride are dissolved in 150 grams of water, the solution cooled and treated with 100 cc of concentrated nitric acid. Dark violet colored crystals of dichlorohexamminomuolnitritodicobaltic nitrate deposit.

$$[(NH_3)_3ClCoOHNO_2CoCl(NH_3)_3]Cl_2 + 2HNO_3 \rightarrow [(NH_3)_3ClCoOHNO_2CoCl(NH_3)_3](NO_3)_2 + 2HCl$$

A. Werner, E. Bindschedler and E. Welti, Ann., **375**, 126 (1910) **20**

$[Co_2(NH_3)_6O_2Cl_2NH_2]Cl_2$

HNO_3 **III-683**

Symmetrical dichlorohexamminomuaminoperoxodicobaltic chloride is triturated several times with concentrated nitric acid, the residue washed with small volumes of water and dried. The compound formed is symmetrical dichlorohexamminomuaminoperoxodicobaltic nitrate.

$$[(NH_3)_3ClCoNH_2O_2CoCl(NH_3)_3]Cl_2 + 2HNO_3 \rightarrow [(NH_3)_3ClCoNH_2O_2CoCl(NH_3)_3](NO_3)_2 + 2HCl$$

A. Werner and M. Grigorieff, Ann., **375**, 108 (1910) *20*

$[Co_2(NH_3)_{10}(NO_2)_4]Cl_2$

$K_4Fe(CN)_6$ **III-684**

When a solution of xantho cobaltic chloride is treated with potassium ferrocyanide, xantho cobaltic ferrocyanide is formed.

$$[Co_2(NH_3)_{10}(NO_2)_4]Cl_2 + K_4Fe(CN)_6 \rightarrow [Co_2(NH_3)_{10}(NO_2)_2]Fe(CN)_6 + 2KCl + 2KNO_2$$

W. Gibbs, Ber., **3**, 43 (1870) *11*

$[Co_2(NH_3)_6O_2Cl_2NH_2]Cl_2$

NH_3 **III-685**

H_2SO_4

Unsymmetrical dichlorohexamminomuaminoperoxodicobaltic chloride is added to liquid ammonia and it dissolves to a dirty brown colored solution. Evaporation of the ammonia leaves a greenish brown residue which is dissolved in water and treated with a dilute solution of sulfuric acid. Greenish black crystals of octamminomuaminoperoxodicobaltic sulfate are formed. The compound is insoluble in water.

(a) $[(NH_3)_2Cl_2CoNH_2O_2Co(NH_3)_4]Cl_2 + 2NH_3 \rightarrow [(NH_3)_4CoNH_2O_2Co(NH_3)_4]Cl_4$

(b) $[(NH_3)_4CoNH_2O_2Co(NH_3)_4]Cl_4 + 2H_2SO_4 \rightarrow [(NH_3)_4CoNH_2O_2Co(NH_3)_4](SO_4)_2 + 4HCl$

A. Werner and M. Grigorieff, Ann., **375**, 113 (1910) *20*

$[Co_2(NH_3)_6O_2Cl_2NH_2]Cl_2$

NH_4Br **III-686**

An aqueous solution of unsymmetrical dichlorohexamminomuaminoperoxodicobaltic chloride is treated with the calculated amount of ammonium bromide and small dark brown needles of unsymmetrical hexamminomuaminoperoxodicobaltic bromide deposit. These are purified by dissolving in a small volume of water and precipitating with hydrobromic acid.

$$[(NH_3)_2CoNH_2O_2CoCl_2(NH_3)_4]Cl_2 + 2NH_4Br \rightarrow [(NH_3)_2CoNH_2O_2CoCl_2(NH_3)_4]Br_2 + 2NH_4Cl$$

A. Werner and M. Grigorieff, Ann., **375**, 113 (1910) *20*

$[Co_2(NH_3)_{10}(NO_2)_2(NO_3)_2]Cl_2$

$(NH_4)_2C_2O_4$ **III-687**

Flavocobaltic chloride reacts with ammonium oxalate to form ammonium chloride, ammonium nitrate and xantho cobaltic oxalate.

$$[Co_2(NH_3)_{10}(NO_2)_2(NO_3)_2]Cl_2 + 2(NH_4)_2C_2O_4 \rightarrow [Co_2(NH_3)_{10}(NO_2)_2](C_2O_4)_2 + 2NH_4Cl + 2NH_4NO_3$$

W. Gibbs, Ber., **6**, 831 (1873) *11*

$[Co_2(NH_3)_6OHClNO_2Cl]Cl_2$

$NaNO_2$ **III-688**

$C_2H_4O_2$

0.5 Gram of dichlorohexamminomuolnitritodicobaltic chloride and 0.8 gram of sodium nitrite are added to 10 cc of water and 2 cc of acetic acid. The solution is heated over a free flame until an orange colored solution is formed. Concentrated hydrochloric acid is added and red orange needles of hexamminomuoldinitritodicobaltic chloride with one half H_2O deposit.

$$[(NH_3)_3ClCoOHNO_2CoCl(NH_3)_3]Cl_2 + NaNO_2 + HC_2H_3O_2 \rightarrow [(NH_3)_3CoOH(NO_2)_2Co(NH_3)_3]Cl_3 + C_2H_3O_2Na + HCl$$

A. Werner and E. Welti, Ann., **375**, 130 (1910) *20*

$[Co_2(NH_3)_6OHClNO_2Cl]Cl_2$

$NaNO_3$ **III-689**

Dichlorohexamminomuolnitritodicobaltic chloride is dissolved in water and treated with a saturated aqueous solution of sodium nitrate. Dark violet colored crystals of dichlorohexamminomuolnitritodicobaltic nitrate deposit.

$$[(NH_3)_3ClCoOHNO_2CoCl(NH_3)_3]Cl_2 + 2NaNO_3 \rightarrow [(NH_3)_3ClCoOHNO_2CoCl(NH_3)_3](NO_3)_2 + 2NaCl$$

A. Werner, E. Bindschedler and E. Welti, Ann., **375**, 126 (1910) *20*

$[Co_2(NH_3)_6OHClNO_2Cl]Cl_2$

Na_2SO_4 **III-690**

An aqueous solution of dichlorohexamminomuolnitritodicobaltic chloride is treated with a saturated aqueous solution of sodium sulfate. A very voluminous, purple mass precipitates. The precipitate is dried on a porous plate, suspended in hydrochloric acid, filtered and washed free of sulfates with water. The compound is dichlorohexamminomuolnitritodicobaltic sulfate and crystallized with one H_2O.

$$[(NH_3)_3ClCoOHNO_2CoCl(NH_3)_3]Cl_2 + Na_2SO_4 \rightarrow [(NH_3)_3ClCoOHNO_2CoCl(NH_3)_3]SO_4 + 2NaCl$$

A. Werner, E. Bindschedler and E. Welti, Ann., **375**, 127 (1910) *20*

$[Co_2(NH_3)_6(OH)_3]Cl_3$

$C_2H_4O_2$ **III-691**

Hexamminomutrioldicobaltic chloride is dissolved in the smallest volume of water possible and acetic acid added. The solution is heated and concentrated on the water bath to syrupy consistency and red crystals deposit. These are removed by filtration and washed with alcohol and ether. The compound is hexamminomuacetatodioldicobaltic chloride and crystallizes with three H_2O. The compound looses its water of crystallization when heated at 100-110°C.

$$[(NH_3)_3Co(OH)_3Co(NH_3)_3]Cl_3 + HC_2H_3O_2 \rightarrow [(NH_3)_3Co(OH)_2(C_2H_3O_2)Co(NH_3)_3]Cl_3 + H_2O$$

A. Werner and M. Grigorieff, Ann., **375**, 115 (1910) *20*

$[Co_2(NH_3)_6(OH)_3]Cl_3$

$C_2H_4O_2$ **III-692**

$NaNO_2$

10 grams of hexamminomutrioldicobaltic chloride are dissolved in 50 cc water with addition of acetic acid to complete the solution. The solution is filtered and 2 grams of sodium nitrite added with stirring until all nitrite is dissolved. The orange colored solution is warmed to 40–50°C and 30 cc of hydrochloric acid added. Orange colored crystals of hexamminomudiolnitritodicobaltic chloride with one H_2O deposit. These are filtered off, washed with 1–1 hydrochloric acid then with alcohol until free of acid.

$$[(NH_3)_3Co(OH)_3Co(NH_3)_3]Cl_3 + NaNO_2 + HC_2H_3O_2 \rightarrow [(NH_3)_3Co(OH)_2NO_2Co(NH_3)_3]Cl_3 + C_2H_3O_2Na + H_2O$$

A. Werner, A. Grün and E. Bindschedler, Ann., **375**, 121 (1910) *20*

$[Co_2(NH_3)_6NH_2OHO_2]Cl_3$

HCl **III-693**

Hexamminomuolamino-peroxodicobaltic chloride in aqueous solution is treated with concentrated hydrochloric acid and warmed on the water bath. As the solution cools, black crystalline symmetrical dichlorohexamminomuaminoperoxodicobaltic chloride deposits.

$$[(NH_3)_3CoNH_2OHO_2Co(NH_3)_3]Cl_3 + HCl \rightarrow [(NH_3)_3CoNH_2O_2CoCl_2(NH_3)_3]Cl_2 + H_2O$$

A. Werner and A. Grigorieff, Ann., **375**, 107 (1910) *20*

$[Co_2(NH_3)_6(OH)_2NH_2]Cl_3$

HCl **III-694**

A solution of hexamminomuaminodioldicobaltic chloride is converted to trichloroaquohexamminomuaminodicobaltic chloride (melanochloride) when warmed with concentrated hydrochloric acid.

$$[(NH_3)_3CoNH_2OHOHCo(NH_3)_3]Cl_3 + 2HCl \rightarrow [(NH_3)_3H_2OClCoNH_2CoCl_2(NH_3)_3]Cl_2 + H_2O$$

A. Werner, Ann., **375**, 26 (1910) *20*

$[Co_2(NH_3)_6(OH)_2NO_2]Cl_3$

HCl **III-695**

6 Grams of hexamminomudiolnitritodicobaltic chloride are added to a solution of 27 cc concentrated hydrochloric acid and 9 cc of water and heated on the water bath while being stirred. The orange colored chloride changes to brown then reddish brown and finally violet. The solution is allowed to cool and crystals of dichlorohexamminomuolnitritodicobaltic chloride deposit. These are washed with alcohol and ether.

$$[(NH_3)_3Co(OH)_2NO_2Co(NH_3)_3]Cl_3 + HCl \rightarrow [(NH_3)_3ClCoOHNO_2CoCl(NH_3)_3]Cl_2 + H_2O$$

A. Werner, A. Grün and E. Bindschedler, Ann., **375**, 124 (1910) *20*

$[Co_2(NH_3)_6(OH)_2NO_2]Cl_3$

HNO_3 **III-696**

4 Grams of hexamminomudiolnitritodicobaltic chloride are dissolved in 50 cc of water, the solution cooled to a low temperature and 50 cc of nitric acid added without stirring. Crystallization begins with the addition of the first drop of nitric acid. The crystals of hexamminomudiolnitritodicobaltic nitrate are washed with hydrochloric acid 1:1 and then with alcohol until free of acid.

$$[(NH_3)_3Co(OH)_2NO_2Co(NH_3)_3]Cl_3 + 3HNO_3 \rightarrow [(NH_3)_3Co(OH)_2NO_2Co(NH_3)_3](NO_3)_3 + 3HCl$$

A. Werner, A. Grün and E. Bindschedler, Ann., **375**, 123 (1910) *20*

$[Co_2(NH_3)_6(OH)_2NO_2]Cl_3$

Na_2SO_4 **III-697**

A solution of 4 grams of hexamminomudiolnitritodicobaltic chloride is filtered and to the filtrate is added a solution of 4 grams of crystalline sodium sulfate. A thick crystalline mass separates and an additional 4 grams of sodium sulfate is added. The orange red crystalline mass is washed with cold water. The compound is hexamminomudiolnitritodicobaltic sulfate and crystallizes with two H_2O.

$$2[(NH_3)_3Co(OH)_2NO_2Co(NH_3)_3]Cl_3 + 3Na_2SO_4 \rightarrow [(NH_3)_3Co(OH)_2NO_2Co(NH_3)_3]_2(SO_4)_3 + 6NaCl$$

A. Werner, A. Grün and E. Bindschedler, Ann., **375**, 123 (1910) *20*

$[Co_2(NH_3)_8H_2ONH_2Cl]Cl_4$

HCl **III-698**

A saturated solution of chloroaquooctamminomuaminodicobaltic chloride is treated with half its volume of concentrated hydrochloric acid while warming on the water bath until the precipitate which first forms redissolves. The solution is cooled and the crystals of chloropentamminocobaltic chloride filtered off. Chlorotetramminocobaltic chloride remains in solution.

$$[(NH_3)_4ClCoNH_2CoH_2O(NH_3)_4]Cl_4 + HCl \rightarrow [CoCl(NH_3)_5]Cl_2 + [CoCl_2(NH_3)_4]Cl + (H_2O)$$

A. Werner, Ann., **375**, 46 (1910) *20*

$[Co_2(NH_3)_8H_2ONH_2Cl]Cl_4$

HNO_3 **III-699**

3 Grams of chloroaquooctamminomuaminodicobaltic chloride are dissolved in 50 cc of ice cold water and the filtered solution is then treated with 15 cc of concentrated nitric acid. Reddish violet crystals of chloronitratooctamminomuaminodicobaltic nitrate are deposited. These are washed with nitric acid (1:1). The salt is easily soluble in water and no immediate precipitate is formed upon the addition of silver nitrate solution.

$$[(NH_3)_4ClCoNH_2CoH_2O(NH_3)_4]Cl_4 + 4HNO_3 \rightarrow [(NH_3)_4ClCoNH_2CoNO_3(NH_3)_4](NO_3)_3 + 4HCl + H_2O$$

A. Werner, Ann., **375**, 49 (1910) *20*

$[Co_2(NH_3)_8(OH)_2]Cl_4$

H_2PtCl_6 **III-700**

2 Grams of octamminomudioldicobaltic chloride are dissolved in 25 cc of water and treated with a very dilute solution of chloroplatinic acid. After a short time, copper red crystals of octamminomudioldicobaltic chloroplatinate form. They are washed with alcohol and ether. The crystals have six H_2O.

$$[(NH_3)_4CoOHOHCo(NH_3)_4]Cl_4 + 2H_2PtCl_6 \rightarrow [(NH_3)_4CoOHOHCo(NH_3)_4](PtCl_6)_2 + 4HCl$$

A. Werner, Ann., **375**, 82 (1910) *20*

$[Co_2(NH_3)_8H_2ONH_2Cl]Cl_4$

H_2SO_4 **III-701**

A freshly prepared solution of chloroaquooctamminomuaminodicobaltic chloride is treated with dilute sulfuric acid and chloroaquooctamminomuaminodicobaltic sulfate is precipitated quantitatively. It is a reddish violet compound which crystallizes with one H_2O and is insoluble in water.

$$[(NH_3)_4H_2OCoNH_2CoCl(NH_3)_4]Cl_4 + 2H_2SO_4 \rightarrow \underline{[(NH_3)_4H_2OCoNH_2CoCl(NH_3)_4](SO_4)_2} + 4HCl$$

A. Werner, Ann., **375**, 47 (1910) *41* *20*

$[Co_2(NH_3)_8NH_2OH]Cl_4$

H_2SO_4 **III-702**

3 Grams of octamminomuolaminodicobaltic chloride are dissolved in 10 cc of water and 2 grams of concentrated sulfuric acid added. The solution is warmed over a free flame until a clear violet colored solution results. As the solution cools, purple violet prisms of octamminomuaminosulfatodicobaltic acid sulfate are deposited. These may be recrystallized from a dilute solution of sulfuric acid.

$$[(NH_3)_4CoNH_2OHCo(NH_3)_4]Cl_4 + 3H_2SO_4 \rightarrow [(NH_3)_4CoNH_2SO_4Co(NH_3)_4]SO_4 \cdot HSO_4 + 4HCl + H_2O$$

A. Werner, Ann., **375**, 52 (1910) *20*

$[Co_2(NH_3)_8O_2NH_2]Cl_4$

H_2SO_4 **III-703**

An aqueous solution of octamminomuaminoperoxodicobaltic chloride is treated with dilute sulfuric acid and octamminomuaminoperoxodicobaltic sulfate precipitates quantitatively as greenish gray silvery prismatic crystals with two H_2O.

$$[(NH_3)_4CoNH_2O_2Co(NH_3)_4]Cl_4 + 2H_2SO_4 \rightarrow [(NH_3)_4CoNH_2O_2Co(NH_3)_4](SO_4)_2 + 4HCl$$

A. Werner, Ann., **375**, 67 (1910) *20*

$[Co_2(NH_3)_8(OH)_2]Cl_4$

$KAuCl_4$ **III-704**

2 Grams of octamminomudioldicobaltic chloride are dissolved in 25 cc of water and a concentrated aqueous solution of potassium chloroaurate added. Cherry red crystals of octamminomudioldicobaltic chloroaurate form. They have four H_2O.

$$[(NH_3)_4CoOHOHCo(NH_3)_4]Cl_4 + 4KAuCl_4 \rightarrow [(NH_3)_4CoOHOHCo(NH_3)_4] \cdot (AuCl_4)_4 + 4KCl$$

A. Werner, Ann., **375**, 82 (1910) *20*

$[Co_2(NH_3)_8(OH)_2]Cl_4$

KI **III-705**

2 Grams of octamminomudioldicobaltic chloride are dissolved in 15 cc of water and solid potassium iodide added. Dark violet crystals of octamminomudioldicobaltic iodide precipitate. These are washed with alcohol and ether.

$$[(NH_3)_4CoOHOHCo(NH_3)_4]Cl_4 + 4KI \rightarrow [(NH_3)_4CoOHOHCo(NH_3)_4]I_4 + 4KCl$$

A. Werner, Ann., **375**, 78 (1910) *20*

$[Co_2(NH_3)_8(OH)_2]Cl_4$

K_2PtCl_4 **III-706**

A dilute aqueous solution of octamminomudioldicobaltic chloride is treated with solid potassium chloroplatinite and small crystals of octamminomudioldicobaltic chloroplatinite deposit.

$$[(NH_3)_4CoOHOHCo(NH_3)_4]Cl_4 + 2K_2PtCl_4 \rightarrow [(NH_3)_4CoOHOHCo(NH_3)_4]\cdot(PtCl_4)_2 + 4KCl$$

A. Werner, Ann., **375**, 81 (1910) *20*

$[Co_2(NH_3)_8H_2ONH_2Cl]Cl_4$

NH_4SCN **III-707**

An aqueous solution of chloroaquooctamminomuaminodicobaltic chloride is treated with a saturated aqueous solution of ammonium thiocyanate and small brownish red crystals of chloroaquooctamminomuaminodicobaltic thiocyanate deposit. The crystals are washed with alcohol and ether then dried over sulfuric acid for 24 hours.

$$[(NH_3)_4H_2OCoNH_2CoCl(NH_3)_4]Cl_4 + 4NH_4SCN \rightarrow [(NH_3)_4H_2OCoNH_2CoCl(NH_3)_4](SCN)_4 + 4NH_4Cl$$

A. Werner, Ann., **375**, 48 (1910) *41* *20*

$[Co_2(NH_3)_8(OH)_2]Cl_4$

$NaBrO_3$ **III-708**

2 Grams of octamminomudioldicobaltic chloride are dissolved in 15 cc of water and solid sodium bromate added when ruby red crystals of octamminomudioldicobaltic bromate form. They are washed with alcohol and ether. The compound is soluble in water.

$$[(NH_3)_4CoOHOHCo(NH_3)_4]Cl_4 + 4NaBrO_3 \rightarrow [(NH_3)_4CoOHOHCo(NH_3)_4](BrO_3)_4 + 4NaCl$$

A. Werner, Ann., **375**, 79 (1910) *20*

$[Co_2(NH_3)_8(OH)_2]Cl_4$

$NaClO_3$ **III-709**

A concentrated aqueous solution of octamminomudioldicobaltic chloride is treated with solid sodium chlorate and bright red crystals of octamminomudioldicobaltic chlorate form. They are washed with alcohol and ether. The compound is easily soluble in water and explodes when heated.

$$[(NH_3)_4CoOHOHCo(NH_3)_4]Cl_4 + 4NaClO_3 \rightarrow [(NH_3)_4CoOHOHCo(NH_3)_4](ClO_3)_4 + 4NaCl$$

A. Werner, Ann., **375**, 79 (1910) *20*

$[Co_2(NH_3)_8(OH)_2]Cl_4$

$NaIO_3$ **III-710**

A dilute aqueous solution of octamminomudioldicobaltic chloride is treated with solid sodium iodate when bright violet crystals of octamminomudioldicobaltic iodate are precipitated. The crystals are washed with alcohol and ether. They are insoluble in water.

$$[(NH_3)_4CoOHOHCo(NH_3)_4]Cl_4 + 4NaIO_3 \rightarrow [(NH_3)_4CoOHOHCo(NH_3)_4](IO_3)_4 + 4NaCl$$

A. Werner, Ann., **375**, 80 (1910) *20*

$[Co_2(NH_3)_8H_2ONH_2Cl]Cl_4$

$NaNO_2$
HNO_3

III-711

A concentrated freshly prepared aqueous solution of chloroaquo-octamminomuaminodicobaltic chloride is treated with solid sodium nitrite and warmed on the water bath. After a short time the color of the solution changes from brownish red to orange. When this change occurs, concentrated nitric acid is added and crystals of octamminomuaminonitritodicobaltic nitrate with one H_2O deposit. The crystals are orange colored.

(a) $[(NH_3)_4ClCoNH_2CoH_2O(NH_3)_4]Cl_4 + NaNO_2$
$\rightarrow [(NH_3)_4CoNH_2NO_2Co(NH_3)_4]Cl_4 + H_2O + NaCl$

(b) $[(NH_3)_4CoNH_2NO_2Co(NH_3)_4]Cl_4 + 4HNO_3$
$\rightarrow [(NH_3)_4CoNH_2NO_2Co(NH_3)_4](NO_3)_4 + 4HCl$

A. Werner, Ann., **375**, 55 (1910) *20*

$[Co_2(NH_3)_8NH_2OH]Cl_4$

$NaNO_2$
$HC_2H_3O_2$
HNO_3

III-712

A freshly prepared concentrated aqueous solution of octamminomu-amino-oldicobaltic chloride is acidified with acetic acid, warmed and sodium nitrite added. The solution changes to a yellowish brown color. It is cooled and nitric acid added. In a short time very fine crystals of octamminomuaminonitritodicobaltic nitrate with one H_2O deposit.

(a) $[(NH_3)_4CoNH_2OHCo(NH_3)_4]Cl_4 + NaNO_2 + HC_2H_3O_2$
$\rightarrow [(NH_3)_4CoNH_2NO_2Co(NH_3)_4]Cl_4 + H_2O + C_2H_3O_2Na$

(b) $[(NH_3)_4CoNH_2NO_2Co(NH_3)_4]Cl_4 + 4HNO_3$
$\rightarrow [(NH_3)_4CoNH_2NO_2Co(NH_3)_4](NO_3)_4 + 4HCl$

A. Werner, Ann., **375**, 55 (1910) *20*

$[Co_2(NH_3)_8(OH)_2]Cl_4$

$Na_2C_2O_4$ **III-713**

An aqueous solution of octamminomudioldicobaltic chloride is treated with solid sodium oxalate when ruby red crystals of octamminomudioldicobaltic oxalate with two H_2O form. They are washed with alcohol and ether. They are insoluble in water.

$$[(NH_3)_4CoOHOHCo(NH_3)_4]Cl_4 + 2Na_2C_2O_4 \rightarrow [(NH_3)_4CoOHOHCo(NH_3)_4](C_2O_4)_2 + 4NaCl$$

A. Werner, Ann., **375**, 80 (1910) *20*

$[Co_2(NH_3)_8(OH)_2]Cl_4$

Na_2CrO_4 **III-714**

A dilute aqueous solution of octamminomudioldicobaltic chloride is treated with solid sodium chromate and light brown crystals of octamminomudioldicobaltic chromate are formed. They are washed with alcohol and ether, then dried.

$$[(NH_3)_4CoOHOHCo(NH_3)_4]Cl_4 + 2Na_2CrO_4 \rightarrow [(NH_3)_4CoOHOHCo(NH_3)_4](CrO_4)_2 + 4NaCl$$

A. Werner, Ann., **375**, 81 (1910) *20*

$[Co_2(NH_3)_8(OH)_2]Cl_4$

Na_2HPO_4 **III-715**

A dilute aqueous solution of octamminomudioldicobaltic chloride is treated with solid dibasic sodium phosphate and rose red crystals of octamminomudioldicobaltic phosphate, dibasic, are precipitated. The crystals are washed with alcohol and ether. They contain six H_2O.

$$[(NH_3)_4CoOHOHCo(NH_3)_4]Cl_4 + 2Na_2HPO_4 \rightarrow [(NH_3)_4CoOHOHCo(NH_3)_4](HPO_4)_2 + 4NaCl$$

A. Werner, Ann., **375**, 81 (1910) *20*

$[Co_2(NH_3)_8H_2ONH_2Cl]Cl_4$

$Na_2S_2O_6$ **III-716**

A freshly prepared solution of chloroaquooctamminomuaminodicobaltic chloride is treated with an aqueous solution of sodium dithionate and insoluble chloroaquooctamminomuaminodicobaltic dithionate precipitates as a voluminous mass of gray violet crystals.

$$[(NH_3)_4H_2OCoNH_2CoCl(NH_3)_4]Cl_4 + 2Na_2S_2O_6 \rightarrow [(NH_3)_4H_2OCoNH_2CoCl(NH_3)_4](S_2O_6)_2 + 4NaCl$$

A. Werner, Ann., **375**, 48 (1910) *20*

$[Co_2(NH_3)_8(OH)_2]Cl_4$

$Na_2S_2O_6$ **III-717**

NH_4Br

150 Grams of octamminomudioldicobaltic chloride are dissolved in the smallest possible quantity of water and treated with sodium dithionate. Yellow crystals of the dithionate are obtained, which are dissolved in water and treated with ammonium bromide which converts the salt to the bromide which is filtered on a porous plate by suction, and purified by alcohol and ether. The product is octamminomudiaminodicobaltic bromide.

(a) $$[(NH_3)_4Co(OH)_2Co(NH_3)_4]Cl_4 + 2Na_2S_2O_6 \rightarrow 4NaCl + [(NH_3)_4Co(OH)_2Co(NH_3)_4](S_2O_6)_2$$

(b) $$[(NH_3)_4Co(OH)_2Co(NH_3)_4](S_2O_6)_2 + 4NH_4Br \rightarrow [(NH_3)_4Co(NH_2)_2Co(NH_3)_4]Br_4 + (NH_4)_2SO_4 + 2H_2SO_3 + H_2SO_4$$

A. Werner, Ann., **375**, 87 (1910) *41*

$[Co_2(NH_3)_8(OH)_2]Cl_4$

Na_2SiF_6 **III-718**

2 Grams of octamminomudioldicobaltic chloride are dissolved in 30 cc of water and treated with a solution of sodium fluosilicate. Bright violet crystals of octamminomudioldicobaltic fluosilicate form. The compound is insoluble in water.

$$[(NH_3)_4CoOHOHCo(NH_3)_4]Cl_4 + 2Na_2SiF_6 \rightarrow [(NH_3)_4CoOHOHCo(NH_3)_4](SiF_6)_2 + 4NaCl$$

A. Werner, Ann., **375**, 83 (1910) *20*

$[Co_2(NH_3)_8H_2ONH_2Cl]Cl_4$

Decomp. **III-719**

When a concentrated aqueous solution of chloroaquooctamminomuaminodicobaltic chloride is allowed to stand for some time, reddish brown prismatic needles of the difficultly soluble octamminomuaminooldicobaltic chloride deposit.

$$[(NH_3)_4H_2OCoNH_2CoCl(NH_3)_4]Cl_4 \rightarrow [(NH_3)_4CoNH_2OHCo(NH_3)_4]Cl_4 + HCl$$

A. Werner, Ann., **375**, 45 (1910) *20*

$[Co_2(NH_3)_{10}NH_2]Cl_5$

HCl **III-720**

Decamminomuaminodicobaltic chloride is digested with a solution of hydrochloric acid in sulfuric acid on the water bath. Hexamminocobaltic chloride and chloropentamminocobaltic chloride are formed.

$$[(NH_3)_5CoNH_2Co(NH_3)_5]Cl_5 + HCl \rightarrow [Co(NH_3)_6]Cl_3 + [Co(NH_3)_5Cl]Cl_2$$

A. Werner, Ann., **375**, 8 (1910) *20*

$[Co_2(NH_3)_6AcNH_2OH]I_3$

AgBr **III-721**

1 Gram of hexamminomuaminoolacetatodicobaltic iodide is rubbed with moist silver bromide and the mixture warmed to 40°C. The solution is filtered and alcohol added to the filtrate when bright red crystals of hexamminomuaminoolacetatodicobaltic bromide form. The aqueous solution of this compound is neutral.

$$[(NH_3)_3CoNH_2OH(C_2H_3O_2)Co(NH_3)_3]I_3 + 3AgBr \rightarrow [(NH_3)_3CoNH_2OH(C_2H_3O_2)Co(NH_3)_3]Br_3 + 3AgI$$

A. Werner and J. Furstenberg, Ann., **375**, 101 (1910) *20*

$[Co_2(NH_3)_8OHNH_2OH]I_3$

AgBr **III-722**

1 Gram of hexamminomuaminodioldicobaltic iodide is triturated with 2 grams of freshly prepared silver bromide. The solution is filtered and absolute alcohol added to the filtrate until it is permanently turbid. Brownish red crystals of hexamminomuaminodioldicobaltic bromide deposit as the solution stands.

$$[(NH_3)_3CoNH_2OHOHCo(NH_3)_3]I_3 + 3AgBr \rightarrow [(NH_3)_3CoNH_2OHOHCo(NH_3)_3]Br_3 + 3AgI$$

A. Werner and J. Furstenberg, Ann., **375**, 91 (1910) *20*

$[Co_2(NH_3)_6AcNH_2OH]I_3$

AgCl **III-723**

1 Gram of hexamminomuaminoolacetatodicobaltic iodide is rubbed with moist silver chloride, water added, the solution filtered and hexamminomuaminoolacetatodicobaltic chloride precipitated with alcohol. The compound precipitates as bright red needles, is soluble in water and its aqueous solutions are neutral.

$$[(NH_3)_3CoNH_2OH(C_2H_3O_2)Co(NH_3)_3]I_3 + 3AgCl \rightarrow [(NH_3)_3CoNH_2OH(C_2H_3O_2)Co(NH_3)_3]Cl_3 + 3AgI$$

A. Werner and J. Furstenberg, Ann., **375**, 101 (1910) *20*

$[Co_2(NH_3)_6OHNH_2OH]I_3$

AgCl **III-724**

Hexamminomuaminodioldicobaltic iodide is triturated with moist silver chloride and alcohol added to the filtrate after removal of the silver iodide. Bright red crystals of hexamminomuaminodioldicobaltic chloride with one H_2O are deposited. The compound is easily soluble in water and the solution is neutral in reaction.

$$[(NH_3)_3CoNH_2OHOHCo(NH_3)_3]I_3 + 3AgCl \rightarrow [(NH_3)_3CoNH_2OHOHCo(NH_3)_3]Cl_3 + 3AgI$$

A. Werner and J. Furstenberg, Ann., **375**, 92 (1910) *20*

$[Co_2(NH_3)_6AcNH_2OH]I_3$

AgCl **III-725**

K_2PtCl_4

Hexamminomuaminoolacetatodicobaltic iodide is rubbed with moist silver chloride and a little water added. The solution is filtered and a solution of potassium chloroplatinite added. After a few minutes, bright red lustrous crystals of hexamminomuaminoolacetatodicobaltic chloroplatinite form.

(a) $$[(NH_3)_3CoNH_2OH(C_2H_3O_2)Co(NH_3)_3]I_3 + 3AgCl \rightarrow [(NH_3)_3CoNH_2OH(C_2H_3O_2)Co(NH_3)_3]Cl_3 + 3AgI$$

(b) $$2[(NH_3)_3CoNH_2OH(C_2H_3O_2)Co(NH_3)_3]Cl_3 + 3K_2PtCl_4 \rightarrow [(NH_3)_3CoNH_2OH(C_2H_3O_2)Co(NH_3)_3]_2PtCl_4)_3 + 6KCl$$

A. Werner and J. Furstenberg, Ann., **375**, 102 (1910) *20*

$[Co_2(NH_3)_6AcNH_2OH]I_3$

$AgNO_3$ **III-726**

1 Gram of hexamminomuaminoolacetatodicobaltic iodide is rubbed with 0.8 gram of silver nitrate, a little water added and the solution filtered. Absolute alcohol is added to the filtrate and bright red crystals of hexamminomuaminoolacetatodicobaltic nitrate separate. The compound is soluble in water and the solution is neutral in reaction.

$$[(NH_3)_3CoNH_2OH(C_2H_3O_2)Co(NH_3)_3]I_3 + 3AgNO_3 \rightarrow [(NH_3)_3CoNH_2OH(C_2H_3O_2)Co(NH_3)_3](NO_3)_3 + 3AgI$$

A. Werner and J. Furstenberg, Ann., **375**, 102 (1910) *20*

$[Co_2(NH_3)_6(OH)NH_2OH]I_3$

$AgNO_3$ **III-727**

1 Gram of hexamminomuaminodioldicobaltic iodide is triturated with a concentrated aqueous solution of 0.8 gram of silver nitrate and the silver iodide removed by filtration. Absolute alcohol is added to the filtrate and small red crystals of hexamminomuaminodioldicobaltic nitrate with two H_2O form. The compound is easily soluble in water and the solution is neutral.

$$[(NH_3)_3CoNH_2OHOHCo(NH_3)_3]I_3 + 3AgNO_3 \rightarrow [(NH_3)_3CoNH_2OHOHCo(NH_3)_3](NO_3)_3 + 3AgI$$

A. Werner and J. Furstenberg, Ann., **375**, 92 (1910) *20*

$[Co_2NHO_2en_4]I_3$

NH_4Br

$AgNO_3$ **III-728**

When a solution of tetraethylenediaminomuaminoperoxodicobaltic iodide is treated with ammonium bromide and the calculated amount of silver nitrate, the corresponding red tetraethylenediaminodicobaltic bromide is precipitated, filtered and washed with alcohol.

$$[(C_2H_8N_2)_2CoNHO_2Co(C_2H_8N_2)_2]I_3 + 3NH_4Br + 3AgNO_3 \rightarrow \underline{[(C_2H_8N_2)_2CoNHO_2Co(C_2H_8N_2)_2]Br_3} + 3AgI + 3NH_4NO_3$$

A. Werner, Ann., **375**, 74 (1910) *41*

$[Co_2(NH_3)_4en_4]I_4 \cdot C_2O_4$

AgCl **III-729**

The ammino ethylenediamine complex of cobalt oxalate iodide is treated with freshly precipitated AgCl. The AgI is removed by filtration; the filtrate is concentrated and gradually deposits light yellow crystals.

$$[Co(NH_3)_2 \cdot 2C_2H_4(NH_2)_2]_2I_4 \cdot (C_2O_4) + 4AgCl \rightarrow \underline{4AgI} + \underline{[Co(NH_3)_2 \cdot 2C_2H_4(NH_2)_2]_2Cl_4(C_2O_4)}$$

A. Werner, Ann., **386**, 78 (1912) *41*

$[Co_2(NH_3)_6(OH)_3](NO_3)_3$

$C_2H_4O_2$ **III-730**

Acetic acid is added to a solution of hexamminomutrioldicobaltic nitrate and the solution concentrated on the water bath. Red monoclinic crystals of hexamminomuacetatodioldicobaltic nitrate with two H_2O deposit. These are pressed on a porous plate, washed with alcohol and ether, then dried.

$$[(NH_3)_3Co(OH)_3Co(NH_3)_3](NO_3)_3 + HC_2H_3O_2 \rightarrow [(NH_3)_3Co(OH)_2(C_2H_3O_2)Co(NH_3)_3](NO_3)_3 + H_2O$$

A. Werner and M. Grigorieff, Ann., **375**, 118 (1910) *20*

$[Co_2(NH_3)_6H_2ONO_3NH_2OH](NO_3)_3$

HCl **III-731**

Nitratoaquohexamminomuaminooldicobaltic nitrate is dissolved in cold water and the solution treated with concentrated hydrochloric acid. Needles of nitratoaquohexamminomuaminooldicobaltic chloride with one H_2O separate.

$$[(NH_3)_3H_2OCoNH_2OHCoNO_3(NH_3)_3](NO_3)_3 + 3HCl \rightarrow [(NH_3)_3H_2OCoNH_2OHCoNO_3(NH_3)_3]Cl_3 + 3HNO_3$$

A. Werner and S. Malmgren, Ann., **375**, 95 (1910) *20*

$[Co_2(NH_3)_6(OH)_2O_2](NO_3)_3$

HCl **III-732**

Hexamminomudiolperoxodicobaltic nitrate is dissolved in cold water containing acetic acid. Upon the addition of concentrated hydrochloric acid grayish-black crystals are deposited. The reaction is almost instantaneous if heat is applied. The corresponding complex sulfate may be used as starting material instead of the nitrate.

$$[(NH_3)_3CoO_2(OH)_2Co(NH_3)_3](NO_3)_3 + 4HCl \rightarrow [(NH_3)_3CoO_2(OH)Co(NH_3)_3]Cl_4 + H_2O + 3HNO_3$$

A. Werner, Ann., **375**, 137 (1910) *41*

$[Co_2(NH_3)_8NH_2SO_4](NO_3)_3$

HCl **III-733**

10 Grams of sulfatooctamminomuaminodicobaltic nitrate are added to 150 cc of concentrated hydrochloric acid and the mixture shaken vigorously at room temperature. The sulfato compound dissolves and the solution is allowed to stand 24 hours at the end of which time a mass of crystals are formed. These are filtered off and dried on a porous plate then dissolved in water, filtered, cooled in an ice bath and concentrated hydrochloric acid added. Brownish violet colored needles of chloroaquooctamminomuamino dicobaltic chloride with $4H_2O$ separate. The salt is easily soluble in water and insoluble in organic solvents. A fresh aqueous solution of the compound reacts with platinous chloride to form a copper bronze colored precipitate, with mercuric chloride to form a carmine red precipitate and with potassium iodide to form a red precipitate.

$$[(NH_3)_4CoNH_2SO_4Co(NH_3)_4](NO_3)_3 + 5HCl + H_2O \rightarrow [(NH_3)_4H_2OCoNH_2CoCl(NH_3)_4]Cl_4 + 3HNO_3 + H_2SO_4$$

A. Werner, Ann., **375**, 45 (1910) *20*

$[Co_2NHHNO_3O_2en_4](NO_3)_3$

KBr **III-734**

4 Grams of tetraethylenediaminemuammoniumperoxodicobaltic nitrate are dissolved in 80 cc of water and 12 grams of potassium bromide added. Red crystals of tetraethylenediaminemuammoniumperoxodicobaltic bromide with three H_2O deposit. They are washed with alcohol and ether.

$$[(C_2H_8N_2)_2CoNHHNO_3O_2Co(C_2H_8N_2)_2](NO_3)_3 + 4KBr \rightarrow [(C_2H_8N_2)_2CoNHHBrO_2Co(C_2H_8N_2)_2]Br_3 + 4KNO_3$$

A. Werner, Ann., **375**, 73 (1910) *20*

$[Co_2NH_2O_2en_4](NO_3)_3$

KI **III-735**

$HC_2H_3O_2$

$Na_2S_2O_3$

To a suspension of tetraethylenediaminemuaminoperoxodicobaltic nitrate in acetic acid, KI is added. The crystalline mass formed is treated with warm $Na_2S_2O_3$ until a pale red solution is obtained. The mixture is cooled and the solid is removed by filtration. The solid product consisting of dark red glistening crystals is recrystallized from a solution of NH_4Br.

(a) $[(C_2H_8N_2)_2CoNH_2O_2Co(C_2H_8N_2)_2](NO_3)_3 + 4KI + 5H_2O + HC_2H_3O_2 + 2Na_2S_2O_3 \rightarrow [(C_2H_8N_2)_2CoNH_2OHCo(C_2H_8N_2)_2]I_4 + 4H_2O + 3KNO_3 + 2NaOH + Na_2S_4O_6 + KC_2H_3O_2$

(b) $[(C_2H_8N_2)_2CoNH_2OHCo(C_2H_8N_2)_2]I_4 + 4NH_4Br \rightarrow [(C_2H_8N_2)_2CoNH_2OHCo(C_2H_8N_2)_2]Br_4 + 4NH_4I$

A. Werner, Ann., **375**, 77 (1910) *41*

$[Co_2NH_2O_2en_4](NO_3)_3$

KSCN **III-736**

HNO_3

NH_4Br

Acetic acid and excess potassium thiocyanate are added to a water suspension of tetraethylenediaminomuaminoperoxodicobaltic nitrate. The thiocyanate which precipitates is dissolved in water on the water bath and then the solution is cooled. Concentrated nitric acid is added in small proportions with stirring until no more oxides of nitrogen escape. The solution is cooled and the solid in suspension removed by filtration, dissolved in water, and treated with ammonium bromide. Red crystals of the product precipitate.

$[(C_2H_8N_2)_2Co(NH_2)O_2Co(C_2H_8N_2)_2](NO_3)_3 + 3KSCN + 16HNO_3 + 3NH_4Br \rightarrow [(C_2H_8N_2)_2Co(NH_2)SO_4Co(C_2H_8N_2)_2]Br_3 + 3KNO_3 + 6H_2O + 3CO_2 + 2H_2SO_4 + 15NO + 3NH_4NO_3 + NO_2$

A. Werner, Ann., **375**, 77 (1910) *41*

$[Co_2(NH_3)_6H_2ONO_3NH_2OH](NO_3)_3$

NH_3 **III-737**

4 Grams of nitratoaquohexamminomuaminooldicobaltic nitrate are added to an excess of liquid ammonia and the ammonia allowed to evaporate. The residue is extracted with cold water, the solution slightly acidified with hydrochloric acid and ammonium sulfate added. A reddish colored precipitate forms which is converted to the nitrate, octamminomuaminooldicobaltic nitrate, by treating with ammonium nitrate. The nitrate is dissolved in water and ammonium bromide added when pure octamminomuaminooldicobaltic bromide is deposited as a red crystalline compound with three H_2O.

(a) $[(NH_3)_3H_2OCoNH_2OHCoNO_3(NH_3)_3](NO_3)_3 + 2NH_3 \rightarrow [(NH_3)_4CoNH_2OHCo(NH_3)_4](NO_3)_4 + H_2O$

(b) $[(NH_3)_4CoNH_2OHCo(NH_3)_4](NO_3)_4 + 4NH_4Br \rightarrow [(NH_3)_4CoNH_2OHCo(NH_3)_4]Br_4 + 4NH_4NO_3$

A. Werner and S. Malmgren, Ann., **375**, 94 (1910) *20*

$[Co_2(NH_3)_6H_2ONO_3NH_2OH](NO_3)_3$

NH_3 **III-738**

Nitratoaquohexamminomuamino-oldicobaltic nitrate reacts with liquid ammonia to form octamminomuamino-oldicobaltic nitrate.

$[(NH_3)_3H_2OCoNH_2OHCoNO_3(NH_3)_3](NO_3)_3 + 2NH_3 \rightarrow [(NH_3)_4CoNH_2OHCo(NH_3)_4](NO_3)_4 + H_2O$

A. Werner and S. Malmgren, Ann., **375**, 95 (1910) *20*

$[Co_2(NH_3)_8ClNO_3NH_2](NO_3)_3$

NH_3

$AgNO_3$ **III-739**

Chloronitratooctamminomuaminodicobaltic nitrate is added to liquid ammonia and the compound dissolves to form a brown colored solution. The ammonia is allowed to evaporate and the residue extracted with water on the water bath until no more residue dissolves. The residue is recrystallized from water acidified with acetic acid. Large bluish red crystals of decamminomuaminodicobaltic chloronitrate deposit. These are dissolved in water, a solution of silver nitrate added and decamminomuaminodicobaltic nitrate deposits. These are recrystallized from water containing nitric acid.

$[(NH_3)_4ClCoNH_2CoNO_3(NH_3)_4](NO_3)_3 + 2NH_3 + AgNO_3 \rightarrow [Co_2(NH_3)_{10}NH_2](NO_3)_5 + AgCl$

A. Werner, Ann., **375**, 43 (1910) *20*

$[Co_2(NH_3)_6H_2ONO_3NH_2OH](NO_3)_3$

NH_4Br **III-740**

Nitratoaquohexamminomuaminooldicobaltic nitrate is dissolved in cold water and solid ammonium bromide added. After a short time a mass of flesh colored crystals of nitratoaquohexamminomuamino-oldicobaltic bromide deposits. The crystals have one H_2O.

$$[(NH_3)_3H_2OCoNH_2OHCoNO_3(NH_3)_3](NO_3)_3 + 3NH_4Br \rightarrow [(NH_3)_3H_2OCoNH_2OHCoNO_3(NH_3)_3]Br_3 + 3NH_4NO_3$$

A. Werner and S. Malmgren, Ann., **375**, 95 (1910) *20*

$[Co_2(NH_3)_6H_2O(OH)_2O_2](NO_3)_3$

$(NH_4)_2SO_4$ **III-741**

Hydroxoaquohexamminomuolperoxodicobaltic nitrate is dissolved in water and the solution saturated with ammonium sulfate. An equal volume of acetic acid is added and, after a short time, the ammonium sulfate acid double salt with hydroxoaquohexamminomuolperoxodicobaltic sulfate crystallizes. It has one H_2O.

$$[(NH_3)_3OHCoOHO_2CoH_2O(NH_3)_3](NO_3)_3 + 2(NH_4)_2SO_4 \rightarrow [(NH_3)_3OHCoOHO_2CoH_2O(NH_3)_3]\cdot(SO_4)_2NH_4 + 3NH_4NO_3$$

A. Werner and E. Welti, Ann., **375**, 135 (1910) *20*

$[Co_2NH_2O_2en_4](NO_3)_3$

NaI **III-742**

$Na_2S_2O_3$

$(HC_2H_3O_2)$

When a suspension of tetraethylenediaminemuaminoperoxodicobaltic nitrate contained in 30 cc H_2O is treated with 3–4 cc glacial acetic acid and then 6 grams of sodium iodide added, a thick black crystalline magma is formed of the periodide. An excess of sodium thiosulfate is then added and the solution warmed till a dark red color develops. Upon cooling the greater part of the iodide separates out and is filtered and washed with alcohol and ether. The product is light red in color.

$$[(C_2H_8N_2)_2CoO_2NH_2Co(C_2H_8N_2)_2](NO_3)_3 + 4NaI + HC_2H_3O_2 + 2Na_2S_2O_3 + H_2O \rightarrow [(C_2H_8N_2)_2CoNH_2(OH)Co(C_2H_8N_2)_2]I_4 + 3NaNO_3 + (Na_2S_4O_6 + 2NaOH + NaC_2H_3O_2)$$

A. Werner, Ann., **375**, 76 (1910) *41*

$[Co_2(NH_3)_6(H_2O)_2NH_2OH](NO_3)_4$

$C_2H_4O_2$ **III-743**

KI

A solution of diaquohexamminomuaminooldicobaltic nitrate is warmed with acetic acid on the water bath and an excess of potassium iodide added. As the solution cools, prismatic needles or lustrous plates of hexamminomuolaminoacetatodicobaltic iodide deposit. The compound is easily soluble in water and warm acetic acid. Aqueous solutions are neutral in reaction.

$$[(NH_3)_3H_2OCoNH_2OHCoH_2O(NH_3)_3](NO_3)_4 + HC_2H_3O_2 + 3KI \rightarrow [(NH_3)_3CoNH_2OH(C_2H_3O_2)Co(NH_3)_3]I_3 + 3KNO_3 + HNO_3 + 2H_2O$$

A. Werner and J. Furstenberg, Ann., **375**, 100 (1910) *20*

$[Co_2(NH_3)_8NH_2O_2](NO_3)_4$

$C_2H_8N_2$ **III-744**

4 Grams of octamminomuaminoperoxodicobaltic nitrate are dissolved in 60 cc of water and 25 grams of ethylene diamine in 10% aqueous solution are added. The solution is heated on the water bath until the odor of ammonia becomes faint. The brown colored solution is cooled and nitric acid added until an olive green color is produced. The solution is cooled in ice and crystals separate. These are dissolved in a minimum of water, filtered and treated with nitric acid. The green crystals of tetraethylenediaminomuaminoperoxodicobaltic nitrate which deposit are washed with alcohol and ether then dried.

$$[(NH_3)_4CoNH_2O_2Co(NH_3)_4](NO_3)_4 + 4C_2H_8N_2 \rightarrow [(C_2H_8N_2)_2CoNH_2O_2Co(C_2H_8N_2)_2](NO_3)_4 + 8NH_3$$

A. Werner, Ann., **375**, 70 (1910) *20*

$[Co_2(NH_3)_8NH_2NO_2](NO_3)_4$

C_5H_5N **III-745**

A warm concentrated aqueous solution of octamminomuaminonitritodicobaltic nitrate is treated with pyridine and the double salt with pyridine deposits as flesh colored crystals. These are washed with alcohol and ether then dried over sulfuric acid in a desiccator.

$$[(NH_3)_4CoNH_2NO_2Co(NH_3)_4](NO_3)_4 + C_5H_5N \rightarrow [(NH_3)_4CoNH_2NO_2Co(NH_3)_4](NO_3)_4 \cdot C_5H_5N$$

A. Werner, Ann., **375**, 56 (1910) *20*

$[Co_2(NH_3)_6C_2H_3O_2(H_2O)_2NH_2](NO_3)_4$

HBr **III-746**

A solution of diaquohexamminomuaminoacetatodicobaltic nitrate is cooled and treated with 40% hydrobromic acid. Red crystals of diaquohexamminomuaminoacetatodicobaltic bromide deposit. The compound dissolves in water and the solution has a strong acid reaction.

$$[(NH_3)_3H_2OCoNH_2(C_2H_3O_2)CoH_2O(NH_3)_3](NO_3)_4 + 4HBr \rightarrow [(NH_3)_3H_2OCoNH_2(C_2H_3O_2)CoH_2O(NH_3)_3]Br_4 + 4HNO_3$$

A. Werner and J. Furstenberg, Ann., **375**, 103 (1910) *20*

$[Co_2(NH_3)_6(H_2O)_2NH_2OH](NO_3)_4$

HCl **III-747**

Diaquohexamminomuolaminodicobaltic nitrate is dissolved in warm water and concentrated hydrochloric acid added. As the solution cools, violet crystals of trichloroaquohexamminomuaminodicobaltic chloride separate. These are converted to tetrachlorohexammino-muaminodicobaltic chloride when heated to 60–5°C.

(a) $[(NH_3)_3H_2OCoNH_2OHCoH_2O(NH_3)_3](NO_3)_4 + 5HCl \rightarrow [(NH_3)_3Cl_2CoNH_2CoClH_2O(NH_3)_3]Cl_2 + 2H_2O + 4HNO_3$

(b) $[(NH_3)_3Cl_2CoNH_2CoClH_2O(NH_3)_3]Cl_2 \rightarrow [(NH_3)_3Cl_2CoNH_2CoCl_2(NH_3)_3]Cl + H_2O$

A. Werner and S. Malmgren, Ann., **375**, 96 (1910) *20*

$[Co_2(NH_3)_8NH_2NO_2](NO_3)_4$

HCl **III-748**

A solution of octamminomuaminonitritodicobaltic nitrate is treated with concentrated hydrochloric acid. The color of the solution changes to dark orange and soon crystallizes. The crystals are removed by filtration, washed with hydrochloric acid and recrystallized from water acidified with hydrochloric acid. Rhombic crystals of octamminomuaminonitritodicobaltic chloride with one H_2O are deposited. The compound is easily soluble in water.

$$[(NH_3)_4CoNH_2NO_2Co(NH_3)_4](NO_3)_4 + 4HCl \rightarrow [(NH_3)_4CoNH_2NO_2Co(NH_3)_4]Cl_4 + 4HNO_3$$

A. Werner, Ann., **375**, 57 (1910) *20*

$[Co_2(NH_3)_6(H_2O)_2NH_2OH](NO_3)_4$

HNO_3 **III-749**

1 Gram of diaquohexamminomuolaminodicobaltic nitrate is added to 7 cc of hot water and dissolved by the addition of a few drops of nitric acid. 18 cc of concentrated nitric acid are added and the color of the solution changes from red to brownish red. After a few minutes, yellowish brown needles begin to form. The solution is cooled in ice and alcohol added to increase the yield. The compound formed is nitratoaquohexamminomuolaminodicobaltic nitrate and the crystals have two H_2O.

$$[(NH_3)_3H_2OCoNH_2OHCoH_2O(NH_3)_3](NO_3)_4 + H_2O \rightarrow [(NH_3)_3H_2OCoNH_2OHCoNO_3(NH_3)_3](NO_3)_3 + H_2O$$

A. Werner and J. Furstenberg, Ann., **375**, 93 (1910) *20*

$[Co_2(NH_3)_8NH_2O_2](NO_3)_4$

HNO_3 **III-750**

$C_2H_8N_2$

When octamminomuaminoperoxodicobaltic nitrate is treated with nitric acid and ethylene diamine, olive-green crystals of tetraethylenediaminemuaminoperoxodicobaltic nitrate are formed.

$$[(NH_3)_4CoNH_2O_2Co(NH_3)_4](NO_3)_4 + 8HNO_3 + 4C_2H_8N_2 \rightarrow [(C_2H_8N_2)_2CoNH_2O_2Co(C_2H_8N_2)_2](NO_3)_4 + 8NH_4NO_3$$

A. Werner, Ann., **375**, 70 (1910) *41*

$[Co_2(NH_3)_8NH_2O_2](NO_3)_4$

HNO_3 **III-751**

$NaNO_2$

A suspension of octamminomuaminoperoxodicobaltic nitrate in water is treated with excess nitric acid. A solution of sodium nitrite is added dropwise until the color changes from green to dirty-brown to orange-yellow. When no more dark spots remain, the product is removed by filtration and recrystallized from dilute nitric acid.

$$4[(NH_3)_4CoNH_2O_2Co(NH_3)_4](NO_3)_4 + 4HNO_3 + 4NaNO_2 \rightarrow 4[(NH_3)_4CoNH_2NO_2Co(NH_3)_4](NO_3)_4 + 4NaNO_3 + 2H_2O + 3O_2$$

A. Werner, Ann., **375**, 54 (1910) *41*

$[Co_2(NH_3)_8NH_2NO_2](NO_3)_4$

H_2SO_4 **III-752**

A solution of octamminomuaminonitritodicobaltic nitrate is treated with dilute sulfuric acid and insoluble octamminomuaminonitritodicobaltic sulfate precipitates as small orange colored crystals with two H_2O. The salt dissolves in concentrated sulfuric acid to form a dark orange colored solution from which water precipitates the compound.

$$[(NH_3)_4CoNH_2NO_2Co(NH_3)_4](NO_3)_4 + 2H_2SO_4 \rightarrow [(NH_3)_4CoNH_2NO_2Co(NH_3)_4](SO_4)_2 + 4HNO_3$$

A. Werner, Ann., **375**, 60 (1910) *20*

$[Co_2(NH_3)_8NH_2O_2](NO_3)_4$

H_2SO_4 **III-753**

H_2O

An aqueous solution of octamminomuaminoperoxodicobaltic nitrate, or the corresponding chloride or acetate, is transformed by the action of dilute sulfuric acid into grayish-green lustrous crystals of dihydrated octamminomuaminoperoxodicobaltic sulfate.

$$[(NH_3)_4CoNH_2O_2Co(NH_3)_4](NO_3)_4 + 2H_2SO_4 + 2H_2O \rightarrow [(NH_3)_4CoNH_2O_2Co(NH_3)_4](SO_4)_2 + 4HNO_3 + 2H_2O$$

A. Werner, Ann., **375**, 67 (1910) *41*

$[Co_2(NH_3)_6(H_2O)_2NH_2OH](NO_3)_4$

KI **III-754**

A dilute solution of diaquohexamminomuolaminodicobaltic nitrate is treated with solid potassium iodide and a crystalline mass precipitates. This is recrystallized from water care being taken to avoid high temperatures as the compound decomposes. If the compound is prepared from too concentrated solutions of the starting material, the nitrate is occluded in the precipitate. The compound formed is hexamminomuaminodioldicobaltic iodide and is obtained as brownish red needles.

$$[(NH_3)_3H_2OCoNH_2OHCoH_2O(NH_3)_3](NO_3)_4 + 3KI \rightarrow [(NH_3)_3CoNH_2OHOHCo(NH_3)_3]I_3 + H_2O + 3KNO_3 + HNO_3$$

A. Werner and J. Furstenberg, Ann., **375**, 91 (1910) *20*

$[Co_2(NH_3)_8NH_2NO_2](NO_3)_4$

KI **III-755**

A cold aqueous solution of octamminomuaminonitritodicobaltic nitrate is treated with a solution of potassium iodide. The brownish red octamminomuaminonitritodicobaltic iodide precipitates. It is recrystallized from water containing potassium iodide and the compound is obtained as large dark red prisms which have one H_2O.

$$[(NH_3)_4CoNH_2NO_2Co(NH_3)_4](NO_3)_4 + 4KI \rightarrow [(NH_3)_4CoNH_2NO_2Co(NH_3)_4]I_4 + 4KNO_3$$

A. Werner, Ann., **375**, 59 (1910) *20*

$[Co_2NH_2O_2en_4](NO_3)_4$

NH_4Br **III-756**

A cold saturated aqueous solution of tetraethylenediaminomuaminoperoxodicobaltic chloride is treated with ammonium bromide and tetraethylenediaminomuaminoperoxodicobaltic bromide precipitates as a crystalline mass which is purified by recrystallization from water by the addition of ammonium bromide. The crystals are yellowish green in transmitted light and almost black in reflected light. Aqueous solutions are yellowish green in color and neutral in reaction.

$$[(C_2H_8N_2)_2CoNH_2O_2Co(C_2H_8N_2)_2](NO_3)_4 + 4NH_4Br \rightarrow [(C_2H_8N_2)_2CoNH_2O_2Co(C_2H_8N_2)_2]Br_4 + 4NH_4NO_3$$

A. Werner, Ann., **375**, 71 (1910) *20*

$[Co_2(NH_3)_6(H_2O)_2NH_2OH](NO_3)_4$

NH_4Br **III-757**

A warm, rather concentrated aqueous solution of diaquohexamminomuolaminodicobaltic nitrate is saturated with ammonium bromide. As the solution cools, dark red crystals of hexamminomuaminodioldicobaltic bromide with one H_2O deposit. The compound is fairly soluble in water and the solution is neutral in reaction.

$$[(NH_3)_3H_2OCoNH_2OHCoH_2O(NH_3)_3](NO_3)_4 + 3NH_4Br \rightarrow [(NH_3)_3CoNH_2OHOHCo(NH_3)_3]Br_3 + 3NH_4NO_3 + HNO_3 + H_2O$$

A. Werner and J. Furstenberg, Ann., **375**, 91 (1910) *20*

$[Co_2(NH_3)_8NH_2O_2](NO_3)_4$

NH_4Cl **III-758**
NH_4Br

1 Gram of octamminomuaminoperoxodicobaltic nitrate and 1 gram of ammonium chloride are dissolved in 5 cc of water and the solution boiled over a free flame until the color of the solution changes to brown. Ammonia is evolved. Solution is filtered and 4–5 volumes of concentrated hydrochloric acid added to the filtrate. After a short time brown to black crystals of hexamminomuolaminoperoxodicobaltic chloride deposit. 1 Gram of these crystals is dissolved in the smallest possible quantity of water and 1.5 grams of silver nitrate added. The solution is heated until no more silver chloride precipitates. Filter and add ammonium bromide. The solution is stirred until green crystals begin to form from the red solution. Addition of alcohol increases the yield.

(a) $[(NH_3)_4CoNH_2O_2Co(NH_3)_4](NO_3)_4 + 3NH_4Cl + H_2O$
$\rightarrow [(NH_3)_3CoNH_2OHO_2Co(NH_3)_3]Cl_3 + NH_3 + 4NH_4NO_3$

(b) $[(NH_3)_3CoNH_2OHO_2Co(NH_3)_3]Cl_3 + 3NH_4Br$
$\rightarrow [(NH_3)_3CoNH_2OHO_2Co(NH_3)_3]Br_3 + 3NH_4Cl$

A. Werner and A. Grigorieff, Ann., **375**, 105 (1910) *20*

$[Co_2(NH_3)_8NH_2O_2](NO_3)_4$

$(NH_4)_2CO_3$ **III-759**
HCl

5 Grams of air dried octamminomuaminoperoxodicobaltic nitrate are suspended in 25 cc of water and 5 grams of ammonium carbonate added. The solution is heated over a free flame until the color changes from olive green to brown red. The solution is filtered and cooled in an ice bath then 20 cc of concentrated hydrochloric acid added dropwise when there is a copious evolution of carbon dioxide and dark olive green crystals begin to deposit. The solution is allowed to stand about 20 minutes then concentrated hydrochloric acid added when crystallization begins. The compound formed is unsymmetrical dichlorohexamminomuaminoperoxodicobaltic chloride and it is soluble in water.

(a) $[(NH_3)_4CoNH_2O_2Co(NH_3)_4](NO_3)_4 + 2(NH_4)_2CO_3$
$\rightarrow [(NH_3)_4CoNH_2O_2Co(NH_3)_4](CO_3)_2 + 4NH_4NO_3$

(b) $[(NH_3)_4CoNH_2O_2Co(NH_3)_4](CO_3)_2 + 6HCl$
$\rightarrow [(NH_3)_4CoNH_2O_2CoCl_2(NH_3)_2]Cl_2 + 2NH_4Cl + 2H_2O + 2CO_2$

A. Werner and M. Grigorieff, Ann., **375**, 111 (1910) *20*

$[Co_2NH_2O_2en_4](NO_3)_4$

NaI **III-760**

$AgNO_3$

3.8 Grams of tetraethylenediaminomuaminoperoxodicobaltic nitrate are suspended in 5 cc of water and 8 cc of ammonium hydroxide added then 8 grams of sodium iodide and a little sodium thiosulfate. The precipitated mass is washed with alcohol and ether then recrystallized from aqueous solution by the addition of potassium iodide. These crystals are dissolved in water and an aqueous solution of 1.8 grams of silver nitrate added. The solution is filtered and 3 grams of silver nitrate added to the filtrate, the solution cooled in ice when crystals of the silver nitrate double salt with tetraethylenediaminomuimino-peroxodicobaltic nitrate precipitates.

(a) $[(C_2H_8N_2)_2CoNH_2O_2Co(C_2H_8N_2)_2](NO_3)_4 + 3NaI$
$\rightarrow [(C_2H_8N_2)_2CoNHO_2Co(C_2H_8N_2)_2]I_3 + 3NaNO_3 + HNO_3$

(b) $[(C_2H_8N_2)_2CoNHO_2Co(C_2H_8N_2)_2]I_3 + 8AgNO_3$
$\rightarrow [(C_2H_8N_2)_2CoNHO_2Co(C_2H_8N_2)_2](NO_3)_3 \cdot 5AgNO_3 + 3AgI$

A. Werner, Ann., **375**, 75 (1910) *20*

$[Co_2(NH_3)_6(H_2O)_4NH_2OH](NO_3)_4$

$NaNO_2$ **III-761**

HCl

1 Gram of tetraaquohexamminomuolaminodicobaltic nitrate is mixed with 0.25 gram of sodium nitrite and sufficient water added to dissolve the material. The solution is warmed and the color changes to orange. When solution is complete it is treated with concentrated hydrochloric acid. Reddish brown crystals of dichlorohexammino-muaminonitritodicobaltic chloride deposit. These have one H_2O.

(a) $[(NH_3)_3(H_2O)_2CoNH_2OHCo(H_2O)_2(NH_3)_3](NO_3)_4 + 5NaNO_2$
$\rightarrow [(NH_3)_3(H_2O)_2CoNH_2NO_2Co(H_2O)_2(NH_3)_3](NO_2)_4 + NaOH$
$+ 4NaNO_3$

(b) $[(NH_3)_3(H_2O)_2CoNH_2NO_2Co(H_2O)_2(NH_3)_3](NO_2)_4 + 4HCl$
$\rightarrow [(NH_3)_3ClCoNH_2NO_2CoCl(NH_3)_3]Cl_2 + 4H_2O + 4HNO_2$

A. Werner and S. Malmgren, Ann., **375**, 99 (1910) *20*

$[Co_2(NH_3)_8NH_2O_2](NO_3)_4$

$Na_2S_2O_6$ **III-762**

An aqueous solution of octamminomuaminoperoxodicobaltic nitrate is acidified with acetic acid and a solution of sodium dithionate added. Lustrous, silky, leafy crystals of octamminomuaminoperoxodicobaltic dithionate with a brown surface color are formed. The compound dissolves in water to produce a green colored solution although it is only slightly soluble in water.

$$[(NH_3)_4CoNH_2O_2Co(NH_3)_4](NO_3)_4 + 2Na_2S_2O_6 \rightarrow [(NH_3)_4CoNH_2O_2Co(NH_3)_4](S_2O_6)_2 + 4NaNO_3$$

A. Werner, Ann., **375**, 70 (1910) *20*

$[Co_2(NH_3)_8NH_2NO_2](NO_3)_4 \cdot C_5H_5N$

H_2SO_4 **III-763**

The pyridine double salt of octamminomuaminonitritodicobaltic nitrate is dissolved in water and a dilute solution of sulfuric acid added. Octamminomuaminonitritodicobaltic sulfate precipitates. The compound has two H_2O.

$$[(NH_3)_4CoNH_2NO_2Co(NH_3)_4](NO_3)_4 \cdot C_5H_5N + 2H_2SO_4 \rightarrow \underline{[(NH_3)_4CoNH_2NO_2Co(NH_3)_4](SO_4)_2} + 4HNO_3 + C_5H_5N$$

A. Werner, Ann., **375**, 57 (1910) *20*

$[Co_2(NH_3)_8(NO_2)_4]SO_4$

$BaCl_2$ **III-764**

Barium chloride reacts with dinitritotetraamminocobaltic sulfate to form dinitritotetraamminocobaltic chloride, and barium sulfate. The complex salt is orange colored.

$$[Co_2(NH_3)_8(NO_2)_4]SO_4 + BaCl_2 \rightarrow \underline{BaSO_4} + [Co_2(NH_3)_8(NO_2)_4]Cl_2$$

W. Gibbs, Ber., **6**, 832 (1873) *11*

$[Co_2(NH_3)_8(NO_2)_4]SO_4$

$Ba(NO_3)_2$ **III-765**

Barium nitrate reacts with dinitrotetraammino cobaltic sulfate to form dinitrotetraamino cobaltic nitrate and barium sulfate. The complex salt is orange colored.

$$[Co_2(NH_3)_8(NO_2)_4]SO_4 + Ba(NO_3)_2 \rightarrow \underline{BaSO_4} + [Co_2(NH_3)_8(NO_2)_4](NO_3)_2$$

W. Gibbs, Ber., **6**, 832 (1873) *11*

$[Co_2(NH_3)_6Cl_2OHNO_2]SO_4$

$Na_2S_2O_6$ **III-766**

Dichlorohexamminomuolnitritodicobaltic sulfate is triturated with a concentrated aqueous solution of sodium dithionate. Violet dichlorohexamminomuolnitritodicobaltic dithionate is formed. The compound is easily soluble in water.

$$[(NH_3)_3ClCoOHNO_2CoCl(NH_3)_3]SO_4 + Na_2S_2O_6 \rightarrow [(NH_3)_3ClCoOHNO_2CoCl(NH_3)_3]S_2O_6 + Na_2SO_4$$

A. Werner, E. Bindschedler and E. Welti, Ann., **375**, 127 (1910) *20*

$[Co_2(NH_3)_8NH_2SO_4]SO_4 \cdot HSO_4$

HNO_3 **III-767**

Octamminomuaminosulfatodicobaltic hydrogen sulfate is suspended in cold water and treated with nitric acid. Octamminomuaminosulfatodicobaltic nitrate separates as a mass of violet red crystals which are recrystallized from water acidified with nitric acid.

$$[(NH_3)_4CoNH_2SO_4Co(NH_3)_4]SO_4 \cdot HSO_4 + 3HNO_3 \rightarrow [(NH_3)_4CoNH_2SO_4Co(NH_3)_4](NO_3)_3 + 2H_2SO_4$$

A. Werner, Ann., **375**, 53 (1910) *20*

$[CoNO_2NCSen_2]_2SO_4$

$Ba(NO_3)_2$ **III-768**

A solution of cis nitritoisothiocyanatodiethylenediamino cobaltic sulfate acidified with acetic acid is treated with the calculated amount of barium nitrate in solution. The solution is warmed on the water bath until no more precipitate forms, filtered and concentrated. Large brown wart shaped crystals of cis nitritoisothiocyanatodiethylenediamino cobaltic nitrate deposit as the solution cools.

$$[NO_2NCSCo(C_2H_8N_2)_2]_2SO_4 + Ba(NO_3)_2 \rightarrow 2[NO_2NCSCo(C_2H_8N_2)_2]NO_3 + \underline{BaSO_4}$$

A. Werner and C. Rix, Ann., **386**, 236 (1912) *20*

$[CoNO_2NCSen_2]_2SO_4$

HBr **III-769**

Cis nitritoisothiocyanatodiethylenediamino cobaltic sulfate is added to concentrated hydrobromic acid and the solution heated to 70–80°C. Gas is evolved and the solution changes to a deep red color. After most of the sulfate has dissolved, the solution is filtered and again heated for a short time then allowed to stand several hours. Dark red crystals of pure cis bromoisothiocyanatodiethylenediamino cobaltic bromide deposit. The yield is good.

$$[NO_2NCSCo(C_2H_8N_2)_2]_2SO_4 + 2HBr \rightarrow 2[BrNCSCo(C_2H_8N_2)_2]Br + H_2SO_4 + 2NO_2$$

A. Werner and R. Schmidt, Ann., **386**, 150 (1912) *20*

$[Co_2(NH_3)_8NH_2NO_2](SO_4)_2$

$BaBr_2$ **III-770**

HBr

A solution of octamminomuaminonitritodicobaltic sulfate is treated with the calculated amount of barium bromide and the solution acidified with hydrobromic acid. This solution is warmed on the water bath, filtered to remove barium sulfate and then acidified with hydrobromic acid until no more precipitation occurs. The solution is concentrated slowly and brown red rhombic prisms of octamminomuaminonitritodicobaltic bromide with three H_2O deposit. The crystals loose their water of crystallization at 105°C.

$$[(NH_3)_4CoNH_2NO_2Co(NH_3)_4](SO_4)_2 + 2BaBr_2 \rightarrow [(NH_3)_4CoNH_2NO_2Co(NH_3)_4]Br_4 + 2BaSO_4$$

A. Werner, Ann., **375**, 58 (1910) *20*

$[Co_2(NH_3)_8NH_2OH](SO_4)_2$

HBr **III-771**

Octamminomuolaminodicobaltic sulfate is treated with concentrated hydrobromic acid. Bromoaquooctamminomuaminodicobaltic bromide separates as a violet brown crystalline mass which is washed with alcohol until free from acid. The residue is dissolved in a small volume of water and the salt precipitated by the addition of hydrobromic acid. The compound dissolves in water to form a reddish brown solution which has an acid reaction. Upon standing, the color changes to carmine red and red prismatic crystals of octamminomuolaminodicobaltic bromide deposit. These are reconverted to bromoaquooctamminomuaminodicobaltic bromide upon addition of hydrobromic acid.

$$[(NH_3)_4CoNH_2OHCo(NH_3)_4](SO_4)_2 + 5HBr \rightarrow \underline{[(NH_3)_4BrCoNH_2CoH_2O(NH_3)_4]Br_4} + 2H_2SO_4$$

A. Werner, Ann., **375**, 50 (1910) *20*

$[Co_2(NH_3)_8NH_2O_2](SO_4)_2$

HBr **III-772**

Octamminomuaminoperoxodicobaltic bromide is formed when octamminomuaminoperoxodicobaltic sulfate is triturated two or three times with concentrated hydrobromic acid. The bromide is recrystallized from water by the addition of hydrobromic acid. It is obtained as dark green crystals with one H_2O. The compound is easily soluble in water.

$$[(NH_3)_4CoNH_2O_2Co(NH_3)_4](SO_4)_2 + 4HBr \rightarrow [(NH_3)_4CoNH_2O_2Co(NH_3)_4]Br_4 + 2H_2SO_4$$

A. Werner, Ann., **375**, 69 (1910) *20*

$[Co_2(NH_3)_8NH_2NO_2](SO_4)_2$

HCl **III-773**

Octamminomuaminonitritodicobaltic sulfate is rubbed with concentrated hydrochloric acid. The sulfate dissolves and, in a short time, the solution sets to a crystalline mass. This mass is dissolved in a small volume of water, filtered and allowed to stand. Dark brown prisms of octamminomuaminonitritodicobaltic chloride with one H_2O deposit. An aqueous solution of the compound is acid in reaction and hydrochloric acid reprecipitates the salt.

$$[(NH_3)_4CoNH_2NO_2Co(NH_3)_4](SO_4)_2 + 4HCl \rightarrow [(NH_3)_4CoNH_2NO_2Co(NH_3)_4]Cl_4 + 2H_2SO_4$$

A.Werner, Ann., **375**, 57 (1910) *20*

$[Co_2(NH_3)_8NH_2O_2](SO_4)_2$

HCl **III-774**

Octamminomuaminoperoxodicobaltic sulfate is triturated two or three times with conc. hydrochloric acid. Octamminomuaminoperoxodicobaltic chloride is formed. It is almost insoluble in concentrated hydrochloric acid but can be recrystallized from a concentrated aqueous solution by the addition of hydrochloric acid. The compound is obtained as small green crystals.

$$[(NH_3)_4CoNH_2O_2Co(NH_3)_4](SO_4)_2 + 4HCl \rightarrow [(NH_3)_4CoNH_2O_2Co(NH_3)_4]Cl_4 + 2H_2SO_4$$

A. Werner, Ann., **375**, 68 (1910) *20*

$[Co_2(NH_3)_8NH_2O_2](SO_4)_2$

HNO_3 **III-775**

Octamminomuaminoperoxodicobaltic sulfate, when treated with nitric acid, yields dark green prismatic crystals of the corresponding complex nitrate. Water solutions of the latter decompose upon heating to yield cobalt oxide.

$$[(NH_3)_4CoNH_2O_2Co(NH_3)_4](SO_4)_2 + 4HNO_3 \rightarrow [(NH_3)_4CoNH_2O_2Co(NH_3)_4](NO_3)_4 + 2H_2SO_4$$

A. Werner, Ann., **375**, 66 (1910) *41*

$[Co_2(NH_3)_6(OH)_2H_2OO_2]_2(SO_4)_3$

NH_4Br **III-776**

Hydroxoaquohexamminomuolperoxodicobaltic sulphate is made into a thick mass with a small volume of water and an excess of solid ammonium bromide added. The mixture is rubbed, dissolved in a small volume of water and excess ammonium bromide added when hydroxoaquohexamminomuolperoxodicobaltic bromide precipitates as a greenish brown crystalline powder with one H_2O. The compound dissolves in water to form a brown solution which is alkaline.

$$[(NH_3)_3OHCoOHO_2CoH_2O(NH_3)_3]_2(SO_4)_3 + 6NH_4Br \rightarrow 2[(NH_3)_3OHCoOHO_2CoH_2O(NH_3)_3]Br_3 + 3(NH_4)_2SO_4$$

A. Werner and E. Welti, Ann., **375**, 136 (1910) *20*

$[Co_2(NH_3)_6(OH)_2H_2OO_2]_2(SO_4)_3$

NH_4Cl **III-777**

1 Gram of hydroxoaquohexamminomuolperoxodicobaltic sulfate is dissolved in a small volume of water acidified with acetic acid and an excess of ammonium chloride added. The mixture is triturated, dissolved in a small volume of water and the solution saturated with ammonium chloride. The precipitate is removed by filtration added to water slightly acidified with acetic acid and warmed until solution is complete. Upon cooling, brown crystals of hydroxoaquohexamminomuolperoxodicobaltic chloride deposit. The compound dissolves in water to form a brown solution.

$$[(NH_3)_3OHCoOHO_2CoH_2O(NH_3)_3]_2(SO_4)_3 + 6NH_4Cl \rightarrow 2[(NH_3)_3OHCoOHO_2CoH_2O(NH_3)_3]Cl_3 + 3(NH_4)_2SO_4$$

A. Werner and E. Welti, Ann., **375**, 136 (1910) *20*

$[Co_2(NH_3)_6(OH)_2H_2OO_2]_2(SO_4)_3$

NH_4NO_3 **III-778**

1 Gram of hydroxoaquohexamminomuolperoxodicobaltic sulfate is dissolved in a small volume of water acidified with acetic acid, solid ammonium nitrate added and the mixture triturated. The solution is filtered and ammonium nitrate added to the filtrate. Hydroxoaquohexamminomuolperoxodicobaltic nitrate precipitates as a crystalline mass. It is recrystallized from warm water by the addition of alcohol. The crystals are dark brown in color.

$$[(NH_3)_3OHCoOHO_2CoH_2O(NH_3)_3]_2(SO_4)_3 + 6NH_4NO_3 \rightarrow 2[(NH_3)_3OHCoOHO_2CoH_2O(NH_3)_3](NO_3)_3 + 3(NH_4)_2SO_4$$

A. Werner and E. Welti, Ann., **375**, 137 (1910) *20*

$[CoH_2ONCSen_2]_2S_2O_6$

KSCN **III-779**

Cis aquoisothiocyanatodiethylenediamino cobaltic dithionate is dissolved in a small volume of water and solid potassium thiocyanate added. A bright red precipitate forms which is dissolved in water to form a concentrated solution and cis aquoisothiocyanatodiethylenediamino cobaltic thiocyanate precipitated by the addition of potassium thiocyanate. The crystals are washed with alcohol and ether.

$$[NCSH_2OCo(C_2H_8N_2)_2]_2S_2O_6 + 2KSCN \rightarrow 2[NCSH_2OCo(C_2H_8N_2)_2](SCN)_2 + K_2S_2O_6$$

A. Werner and R. Schmidt, Ann., **386**, 156 (1912) *20*

$[Co_2(OH)_2en_4](S_2O_6)_2$

NH_4Br **III-780**

5 Grams of tetraethylenediaminomudioldicobaltic dithionate are digested for 10 minutes with 10 grams of ammonium bromide and 25 cc of water. 10 cc of water are added, the solution filtered and solid ammonium bromide added. Red violet crystals of tetraethylenediaminomudioldicobaltic bromide with four H_2O deposit.

$$[(C_2H_8N_2)_2CoOHOHCo(C_2H_8N_2)_2](S_2O_6)_2 + 4NH_4Br \rightarrow [(C_2H_8N_2)_2CoOHOHCo(C_2H_8N_2)_2]Br_4 + (NH_4)_2S_2O_6$$

A. Werner, Ann., **375**, 85 (1910) *41* *20*

$[Co_3(NH_3)_6(OH)_6]Cl_3$

HCl **III-781**

A solution of hexamminohexoltricobaltic chloride is treated with hydrochloric acid in the cold when one of the hydroxo bridges is broken with the formation of diaquohexamminopentoltricobaltic chloride. The compound is obtained as a green mass which dissolves in water to form a greenish brown solution from which the compound may be reprecipitated upon the addition of hydrochloric acid.

$$[(NH_3)_3Co(OH)_3Co(OH)_3Co(NH_3)_3]Cl_3 + HCl + H_2O \rightarrow \underline{[(NH_3)_3H_2OCo(OH)_2H_2OCo(OH)_3Co(NH_3)_3]Cl_4}$$

A. Werner and E. Welti, Ann., **375**, 143 (1910) *20*

$[Co_3(NH_3)_6(OH)_6]Cl_3$

NH_4Br **III-782**

2 Grams of hexamminohexoltricobaltic chloride is treated with 1 cc of concentrated ammonium hydroxide and added to 80–100 cc of cold water. The deep brown colored solution is filtered quickly, cooled and ammonium bromide added to the filtrate. Small brown crystals of hexamminohexoltricobaltic bromide deposit. They are washed with alcohol and ether then dried.

$$[(NH_3)_3Co(OH)_3Co(OH)_3Co(NH_3)_3]Cl_3 + 3NH_4Br \rightarrow [(NH_3)_3Co(OH)_3Co(OH)_3Co(NH_3)_3]Br_3 + 3NH_4Cl$$

A. Werner and E. Welti, Ann., **375**, 142 (1910) *20*

$[Co_3(NH_3)_6(OH)_6]Cl_3$

$Na_2S_2O_6$ **III-783**

1 Gram of hexamminohexoltricobaltic chloride is dissolved in water, filtered and the filtrate treated with solid sodium dithionate. Brown crystals of hexamminohexoltricobaltic dithionate form. The compound is almost insoluble in water.

$$2[(NH_3)_3Co(OH)_3Co(OH)_3Co(NH_3)_3]Cl_3 + 3Na_2S_2O_6 \rightarrow [(NH_3)_3Co(OH)_3Co(OH)_3Co(NH_3)_3]_2(S_2O_6)_3 + 6NaCl$$

A. Werner and E. Welti, Ann., **375**, 143 (1910) *20*

$[Co_3(OH)_4(H_2O)_2en_4](SO_4)_2$

NH_4Br **III-784**

1 Gram of diaquotetradiethylenediamino tetroltricobaltic sulfate is added to 3 grams of ammonium bromide and a little water added. The solution is warmed and it becomes a red violet color. The solution is cooled and solid sodium bromide added. A crystalline mass of trans bromoamminodiethylenediamino cobaltic bromide mixed with ammonium sulfate precipitates. The precipitate is dissolved in hot water, filtered and cooled. Dark red violet to black rhombic crystals of the bromide deposit.

$$[(C_2H_8N_2)_2Co(OH)_2Co(H_2O)_2(OH)_2Co(C_2H_8N_2)_2](SO_4)_2 + 6NH_4Br + 2H_2O \rightarrow 2[BrNH_3Co(C_2H_8N_2)_2]Br_2 + [Co(H_2O)_6]SO_4 + (NH_4)_2SO_4 + 2(NH_4OH)$$

A. Werner and W. Boës, Ann., **386**, 179 (1912) *20*

$[Co_3(NH_3)_6(OH)_6]_2(SO_4)_3$

NH_4Cl **III-785**

24 Grams of hexamminohexoltricobaltic sulfate are mixed with 24 grams of ammonium chloride, a small volume of water added and the mixture rubbed thoroughly. The residue obtained by filtration is retreated with ammonium chloride as above and this process repeated three times. The final residue is the olive gray colored hexamminohexoltricobaltic chloride. It is purified by dissolving 2 grams in 80 cc of water acidified with acetic acid, filtering and adding 2 grams of ammonium chloride to the filtrate. Dark green crystals of the chloride with three H_2O are obtained. Additional chloride may be obtained by cooling the solution and adding hydrochloric acid.

$$[(NH_3)_3Co(OH)_3Co(OH)_3Co(NH_3)_3]_2(SO_4)_3 + 6NH_4Cl \rightarrow 2[(NH_3)_3Co(OH)_3Co(OH)_3Co(NH_3)_3]Cl_3 + 3(NH_4)_2SO_4$$

A. Werner and E. Welti, Ann., **375**, 141 (1910) *20*

H_2CoO_3

H_2O_2 **III-786**

Cobaltous acid is reduced to cobaltous hydroxide by hydrogen peroxide.

$$H_2CoO_3 + H_2O_2 \rightarrow Co(OH)_2 + H_2O + O_2$$

A. C. Robertson, J. Am. Chem. Soc., **48**, 2078 (1926) *1*

H_2CoO_3

H_2O_2 **III-787**

HCl

Cobaltous acid is reduced by hydrogen peroxide in the presence of hydrochloric acid.

$$H_2CoO_3 + H_2O_2 + 2HCl \rightarrow CoCl_2 + 3H_2O + O_2$$

A. C. Robertson, J. Am. Chem. Soc., **48**, 2078 (1926) *1*

$H_3Co(CN)_6$

$BaCO_3$ **III-788**

Cobalticyanic acid is neutralized by barium carbonate and the solution evaporated.

$$2H_3Co(CN)_6 + 3BaCO_3 \rightarrow Ba_3[Co(CN)_6]_2 + (3H_2O + 3CO_2)$$

C. Zwenger, Ann., **62**, 169 (1847) *25*

$H_3Co(CN)_6$

NH_4OH **III-789**

Cobalticyanic acid is neutralized with ammonium hydroxide and the solution evaporated.

$$H_3Co(CN)_6 + 3NH_4OH \rightarrow \underline{(NH_4)_3Co(CN)_6 \cdot H_2O} + 2H_2O$$

C. Zwenger, Ann., **62**, 157 (1847) *25*

$H_3Co(CN)_6$

$NiSO_4$ **III-790**

Cobalticyanic acid is added in excess to a solution of nickelous sulfate.

$$2H_3Co(CN)_6 + 3NiSO_4 + 12H_2O \rightarrow Ni_3(Co(CN)_6)_2 \cdot 12H_2O + (3H_2SO_4)$$

C. Zwenger, Ann., **62**, 157 (1847) *25*

$KCo(CO)_4$

NO **III-791**

H_2O

Cobalt nitrosyl carbonyl is formed by the addition of nitric oxide to the potassium salt of cobalt tetracarbonyl.

$$KCo(CO)_4 + NO + H_2O \rightarrow Co(CO)_3NO + CO + KOH + \tfrac{1}{2}H_2$$

A. A. Blanchard and P. Gilmont, J. Am. Chem. Soc., **62**, 1192 (1940) *16*

$KCo(NO_2)_4 \cdot 2NH_3$

NH_4NO_3 **III-792**

Potassium tetranitrito cobaltate diammoniate is heated with an equivalent weight of ammonium nitrate to give cobaltic nitrite triammoniate.

$$KCo(NO_2)_4 \cdot 2NH_3 + NH_4NO_3 \rightarrow Co(NO_2)_3 \cdot 3NH_3 + (HNO_2 + KNO_3)$$

A. Werner, Helv. Chim., **1**, 9 (1918) *78*

$KH_2Co_3(CN)_{11} \cdot H_2O$

$AgNO_3$ **III-793**

A salmon colored precipitate of silver cobaltocobalti cyanide is formed on adding a large excess of silver nitrate to a solution of monopotassium cobaltocobalti cyanide.

$$KH_2Co_3(CN)_{11} \cdot H_2O + 3AgNO_3 \rightarrow \underline{Ag_3Co_3(CN)_{11} \cdot H_2O} + KNO_3 + 2HNO_3$$

C. L. Jackson and A. M. Comey, Am. Chem. J., **19**, 275 (1897) *13*

$KH_2Co_3(CN)_{11} \cdot H_2O$

$BaCl_2$ **III-794**

Solutions of monopotassium cobaltocobalti cyanide and barium chloride in excess react to form a precipitate of barium cobaltocobalti cyanide.

$$KH_2Co_3(CN)_{11} \cdot H_2O + BaCl_2 + H_2O \rightarrow .5H_2O + \underline{BaHCo_3 \cdot (CN)_{11} \cdot 1.5H_2O} + KCl + HCl$$

C. L. Jackson and A. M. Comey, Am. Chem. J., **19**, 275 (1897) *13*

$KH_2Co_3(CN)_{11} \cdot H_2O$

$CuSO_4$ **III-795**

A greenish blue flocculent precipitate of cupric cobaltocobalti cyanide is formed by adding copper sulfate to monopotassium cobaltocobalti cyanide.

$$2KH_2Co_3(CN)_{11} \cdot 2H_2O + 3CuSO_4 \rightarrow \underline{Cu_3(Co_3(CN)_{11})_2 \cdot 4H_2O} + K_2SO_4 + 2H_2SO_4$$

C. L. Jackson and A. M. Comey, Am. Chem. J., **19**, 276 (1897) *13*

$KH_2Co_3(CN)_{11} \cdot H_2O$

$KC_2H_3O_2$ **III-796**

A pink, flocculent precipitate of dipotassium cobaltocobalti cyanide is produced when a solution of monopotassium cobaltocobalti cyanide is treated with potassium acetate and alcohol.

$$KH_2Co_3(CN)_{11} \cdot H_2O + KC_2H_3O_2 + H_2O \rightarrow \underline{K_2HCo_3(CN)_{11} \cdot 2H_2O} + HC_2H_3O_2$$

C. L. Jackson and A. M. Comey, Am. Chem. J., **19**, 271 (1897)
Ref., J. Am. Chem. Soc., **19**, 103 (1897) *1*
13

$K_2Co(C_2O_4)_2$

H_2O_2 **III-797**

A solution of potassium cobalt oxalate obtained from cobalt and potassium oxalates is treated with 20% solution of hydrogen peroxide.

$$2K_2Co(C_2O_4)_2 + 3H_2O + H_2O_2 \rightarrow \underline{(K_2Co(C_2O_4)_2 \cdot 2H_2O)_2O}$$

R. G. Durrant, J. Chem. Soc. (London), **87**, 1785 (1905) *25*

$K_2Co(NO_2)_4$

$K_2Pb(NO_2)_4$ **III-798**

Potassium cobaltonitrite reacts with potassium plumbonitrite.

$$K_2Co(NO_2)_4 + K_2Pb(NO_2)_4 \rightarrow 2KNO_2 + Pb(Co(NO_2)_6)$$

Cuttica, Gazz. chim. ital., **53**, 185 (1923) *25*

K_2CoO_3

H_2SO_4 **III-799**

When potassium cobaltite is treated with cold dilute sulfuric acid, cobalt oxysulfate is formed.

$$K_2CoO_3 + 2H_2SO_4 \rightarrow K_2SO_4 + CoO_2SO_3 + 2H_2O$$

A. H. McConnell and E. S. Hanes, J. Chem. Soc. (London), **71**:1 588 (1897) *103*

$K_3Co(CN)_6$

$AgNO_3$ **III-800**

Silver nitrate and potassium cobalticyanide react in solution to precipitate silver cobalticyanide.

$$K_3Co(CN)_6 + 3AgNO_3 \rightarrow \underline{Ag_3Co(CN)_6} + 3KNO_3$$

E. H. Miller and J. A. Mathews, J. Am. Chem. Soc., **22**, 63 (1900) *13*

$K_3Co(CN)_6$

$Bi(NO_3)_3$ **III-801**

A solution of potassium cobalticyanide is treated with a nitric acid solution of bismuth nitrate.

$$K_3Co(CN)_6 + Bi(NO_3)_3 + 5H_2O \rightarrow BiCo(CN)_6 \cdot 5H_2O + (3KNO_3)$$

Fischer and Cuntze, Chem. Ztg., **26**, 872 (1902) *25*

$K_3Co(CN)_6$

$CoCl_2$ **III-802**

Cobaltous chloride and potassium cobalticyanide react in solution to form a precipitate of cobaltous cobalticyanide.

$$2K_3Co(CN)_6 + 3CoCl_2 \rightarrow \underline{Co_3(Co(CN)_6)_2} + 6KCl$$

E. H. Miller and J. A. Mathews, J. Am. Chem. Soc., **22**, 67 (1900) *13*

$K_3Co(CN)_6$

CuCl **III-803**

Cuprous chloride and potassium cobalticyanide react in solution to precipitate cuprous cobalticyanide.

$$K_3Co(CN)_6 + 3CuCl \rightarrow 3KCl + \underline{Cu_3Co(CN)_6}$$

E. H. Miller and J. A. Mathews, J. Am. Chem. Soc., **22**, 65 (1900) *13*

$K_3Co(CN)_6$

$CuSO_4$ **III-804**

Copper sulfate and potassium cobalticyanide react in solution to precipitate cupric cobalticyanide.

$$2K_3Co(CN)_6 + 3CuSO_4 \rightarrow \underline{Cu_3(Co(CN)_6)_2} + 3K_2SO_4$$

E. H. Miller and J. A. Mathews, J. Am. Chem. Soc., **22**, 64 (1900) *13*
1

$K_3Co(CN)_6$

$FeCl_3$ **III-805**

Potassium cobalticyanide and ferric chloride react in solution to form ferric cobalticyanide which precipitates on boiling.

$$K_3Co(CN)_6 + FeCl_3 \rightarrow \underline{FeCo(CN)_6} + 3KCl$$

E. H. Miller and J. A. Mathews, J. Am. Chem. Soc., **22**, 65 (1900) *13*

$K_3Co(CN)_6$

$FeSO_4$ **III-806**

Ferrous sulfate and potassium cobalticyanide react in solution to give a yellow, amorphous, slow-settling precipitate, which seems to be a normal ferrous cobalticyanide.

$$2K_3Co(CN)_6 + 3FeSO_4 \rightarrow \underline{Fe_3(Co(CN)_6)_2} + 3K_2SO_4$$

E. H. Miller and J. A. Mathews, J. Am. Chem. Soc., **22**, 66 (1900) *13*

$K_3Co(CN)_6$

HNO_3 **III-807**

A solution of potassium cobalticyanide is evaporated with an excess of nitric acid and the residue extracted with water.

$$K_3Co(CN)_6 + 3HNO_3 \rightarrow H_3Co(CN)_6 + 3KNO_3$$

C. Zwenger, Ann., **62**, 157 (1847) *25*

$K_3Co(CN)_6$

HNO_3 **III-808**

A red compound is obtained when strong solutions of potassium cobalticyanide and nitric acid are boiled together.

$$3K_3Co(CN)_6 + 9HNO_3 + H_2O$$
$$\rightarrow KH_2Co_3(CN)_{11} \cdot H_2O + 8KNO_3 + 6HCN + NO_2 + HCNO$$

C. L. Jackson and A. M. Comey, Am. Chem. J., **19**, 271 (1897)
Ref., J. Am. Chem. Soc., **19**, 103 (1897) *1*

$K_3Co(CN)_6$

H_2SO_4 **III-809**

Potassium cobalticyanide, on hydrolysis with concentrated sulfuric acid, yields cobaltous sulfate and carbon dioxide. With more dilute acid, the hydrolysis is slow but on prolonged refluxing proceeds according to the following equations:

(a) $$2K_3Co(CN)_6 + 11H_2SO_4 + 13H_2O$$
$$\rightarrow 3K_2SO_4 + 11CO + 6(NH_4)_2SO_4 + 2CoSO_4 + CO_2$$

(b) $$2K_3Co(CN)_6 + 6H_2SO_4 + 2H_2O$$
$$\rightarrow 3K_2SO_4 + 2CoSO_4 + (NH_4)HSO_4 + 11HCN + CO_2$$

Bassett, Chem. and Ind., **43**, 386 (1924) *63*

$K_3Co(CN)_6$

$HgNO_3$ **III-810**

Mercurous nitrate and potassium cobalticyanide react in solution to precipitate mercurous cobalticyanide.

$$K_3Co(CN)_6 + 3HgNO_3 \rightarrow 3KNO_3 + \underline{Hg_3Co(CN)_6}$$

E. H. Miller and J. A. Mathews, J. Am. Chem. Soc., **22**, 64 (1900) *13*

$K_3Co(CN)_6$

$ZnSO_4$ **III-811**

Zinc cobalticyanide is formed as a pure, white easily filtered precipitate when a solution of zinc sulfate is added to a solution of potassium cobalticyanide.

$$2K_3Co(CN)_6 + 3ZnSO_4 \rightarrow \underline{Zn_3(Co(CN)_6)_2} + 3K_2SO_4$$

E. H. Miller and J. A. Mathews, J. Am. Chem. Soc., **22**, 67 (1900) *13*

$K_3Co(CN)_6$

Light **III-812**

Solutions of potassium cobalticyanide turn yellow when exposed to light due to the following reactions:

(a) $K_3Co(CN)_6 + H_2O \rightarrow K_3Co(CN)_5OH + HCN$

(b) $K_3Co(CN)_6 + H_2O \rightarrow K_2Co(CN)_5 \cdot H_2O + KCN$

R. Schwarz and K. Tede, Ber., **60**, 69 (1927)
Ref., Mathieu and Cornevin, J. Chim. phys., **36**, 271 (1939) *69*

$K_3Co(C_2O_4)_3$

NaCl **III-813**

To a solution of potassium cobalt oxalate which had been saturated at 30°, with the same amount of sodium chloride, alcohol is added. Dark green crystals of potassium cobalt sodium oxalate are obtained.

$$K_3Co(C_2O_4)_3 + 2NaCl + 4H_2O \rightarrow \underline{KNa_2Co(C_2O_4)_3} \cdot 4H_2O + 2KCl$$

F. Kehrmann and N. Pickersgill, Z. anorg. Chem., **4**, 133 (1893) *28*

$K_3Co(C_2O_4)_3$

Light **III-814**

The photochemical decomposition of potassium cobaltioxalate was studied:

(a) $2K_3Co(C_2O_4)_3 \rightarrow 2K_2Co(C_2O_4)_2 + K_2C_2O_4 + 2CO_2$

(b) $K_2Co(C_2O_4)_2 \rightleftarrows 2K^+ + Co(C_2O_4)_2^-$

(c) $Co(C_2O_4)_2^- \rightleftarrows Co^{++} + 2C_2O_4^-$

J. Vranek, Z. Elektrochem., **23**, 338 (1917) *86*

$K_3[Co(NO_2)_6]$

$C_3H_{10}N_2$ **III-815**

10 Grams of trimethylenediamine are diluted with 2–3 times its volume of water, the calculated amount of potassium cobaltic nitrite added and the solution heated on the water bath. When the reaction is complete the solution is cooled and large compact yellowish brown crystals of trans dinitritoditrimethylenediamino cobaltic nitrite deposit.

A solution of 1 gram of the compound in 20 cc of water reacts with concentrated sulfuric acid when heated. Gas is evolved and a red solution results. Nitric acid causes the evolution of gas and forms a violet solution. Concentrated hydrobromic acid produces a green solution. Concentrated potassium hydroxide produces a black precipitate and the following reagents give yellow crystalline precipitates: auric chloride, platinous chloride, potassium dichromate, mercuric chloride, ammonium thiocyanate, potassium bromide and potassium iodide.

$$K_3[Co(NO_2)_6] + 2C_3H_{10}N_2 \rightarrow [(NO_2)_2Co(C_3H_{10}N_2)_2]NO_2 + 3KNO_2$$

A. Werner and G. Lindenberg, Ann., **386**, 265 (1912) *20*

$K_4Co(CN)_6$

NaOCl **III-816**

Potassium cobaltocyanide and sodium hypochlorite react in solution to form potassium cobalticyanide.

$$2K_4Co(CN)_6 + NaOCl + H_2O \rightarrow 2K_3Co(CN)_6 + 2KOH + NaCl$$

E. H. Miller and J. A. Mathews, J. Am. Chem. Soc., **22**, 63 (1900) *13*

$K_4Co(CN)_6$

O_2 **III-817**

If potassium cobaltocyanide solution is evaporated to dryness in contact with the air it oxidizes to the cobaltic state according to the following equation:

$$2K_4Co(CN)_6 + O \rightarrow 2K_3Co(CN)_6 + K_2O$$

Jean A. Sanchez, Bull. Soc. Chim., (France), (4), **5**, 642 (1909) *31*

NH_4CoPO_4

Δ **III-818**

Ammonium cobaltous orthophosphate is ignited.

$$2NH_4CoPO_4 \rightarrow Co_2P_2O_7 + (H_2O + 2NH_3)$$

Clarke and Lewis, Am. J. Sci. (Sill) [3], **14**, 281 (1877) *26*

$Na_3Co(NO_2)_6$

$AgNO_3$ **III-819**

KCl

A yellow precipitate of potassium cobaltinitrite is formed when a potassium chloride solution is treated with sodium cobaltinitrite. This is converted into the corresponding silver salt in presence of silver nitrate.

(a) $Na_3Co(NO_2)_6 + (3KCl) \rightarrow \underline{K_3Co(NO_2)_6} + (3NaCl)$

(b) $K_3Co(NO_2)_6 + 3AgNO_3 \rightarrow Ag_3Co(NO_2)_6 + (3KNO_3)$

L. L. Burgess and O. Kamm, J. Am. Chem. Soc., **34**, 653 (1912) *1*

$Na_3Co(NO_2)_6$

CH_3NH_3Cl **III-820**

$C_2H_5NH_3Cl$

From a nearly saturated solution of methylammonium chloride, an excess of saturated solution of sodium cobaltinitrite precipitates yellow crystalline methylammonium cobaltinitrite. Ethylammonium cobaltinitrite is prepared in a similar manner.

(a) $Na_3Co(NO_2)_6 + 3CH_3NH_3Cl$
$\rightarrow \underline{(CH_3NH_3)_3Co(NO_2)_6} + 3NaCl$

(b) $Na_3Co(NO_2)_6 + 3C_2H_5NH_3Cl$
$\rightarrow (C_2H_5NH_3)_3Co(NO_2)_6 + 3NaCl$

M. Cunningham and F. M. Perkin, J. Chem. Soc. (London), **95:2** 1564 (1909) *57*

$Na_3Co(NO_2)_6$

KCl **III-821**

When an excess potassium salt is added to sodium cobaltinitrite, tripotassium cobaltinitrite is precipitated. In an excess of sodium cobaltinitrite, dipotassium cobaltinitrite is formed.

(a) $Na_3Co(NO_2)_6 + 3KCl \rightarrow \underline{K_3Co(NO_2)_6} + 3NaCl$

(b) $Na_3Co(NO_2)_6 + 2KCl \rightarrow K_2NaCo(NO_2)_6 + 2NaCl$

M. Cunningham and F. M. Perkin, J. Chem. Soc. (London), **95:2** 1563 (1909) *57*

$Na_3Co(NO_2)_6$

Tl_2CO_3 **III-822**

Scarlet thallium cobaltinitrite is precipitated by mixing solutions of sodium cobaltinitrite and thallium carbonate.

$$2Na_3Co(NO_2)_6 + 3Tl_2CO_3 \rightarrow \underline{2Tl_3Co(NO_2)_6} + (3Na_2CO_3)$$

M. Cunningham and F. M. Perkin, J. Chem. Soc. (London), **95:2** 1569 (1909) *57*

Cb

Cl_2 **III-823**

Columbium is heated and chlorine is passed over it. The pentachloride is formed quantitatively.

$$2Cb + 5Cl_2 \rightarrow 2CbCl_5$$

W. v. Bolton, Z. Elektrochem., **13**, 149 (1907) *86*

Cb

NH_3 **III-824**

Filings of the metal are heated in an atmosphere of ammonia and air, and the nitride is formed, (a), which, when brought into contact with alkali or water hydrolyzes rapidly (b).

(a) $(12Cb + 20NH_3 + 15O_2 \rightarrow 4Cb_3N_5 + 30H_2O)$

(b) $Cb_3N_5 + 15KOH \rightarrow 5NH_3 + 3K_5CbO_5$

W. v. Bolton, Z. Elektrochem., **13**, 148 (1907) *86*

Cb

S **III-825**

Columbium and sulfur are heated together in a crucible, covered with KCl; soon a strongly exothermic reaction starts, whereby the whole mass becomes glowing and the sulfide is formed.

$$2Cb + 5S \rightarrow Cb_2S_5$$

W. v. Bolton, Z. Elektrochem., **13**, 149 (1907) *86*

Cb

Se **III-826**

Columbium and selenium are heated together in a crucible, covered with KCl. Soon a strongly exothermic reaction starts, whereby the whole mass becomes glowing and the selenide is formed.

$$2Cb + 5Se \rightarrow Cb_2Se_5$$

W. v. Bolton, Z. Elektrochem., **13**, 149 (1907) *86*

$CbAl_3$

HCl **III-827**

Columbium aluminide dissolves upon long boiling with concentrated hydrochloric acid.

$$(CbAl_3 + 14HCl \rightarrow CbCl_5 + 3AlCl_3 + 7H_2)$$

Marignac, Compt. rend., **66**, 182 (1868) *29*

$CbAl_3$

HF **III-828**

Columbium aluminide is attacked by hydrofluoric acid.

$$(CbAl_3 + 14HF \rightarrow CbF_5 + 3AlF_3 + 7H_2)$$

Marignac, Compt. rend., **66**, 182 (1868) *29*

$CbAl_3$

O_2 **III-829**

Columbium aluminide oxidizes rather incompletely upon roasting.

$$(2CbAl_3 + 7O_2 \rightarrow Cb_2O_5 + 3Al_2O_3)$$

Marignac, Compt. rend., **66**, 182 (1868) *29*

CbH

HF **III-830**

Columbium hydride is attacked and dissolved by hydrofluoric acid.

$$[CbH + 5HF \rightarrow CbF_5 + 3H_2]$$

Marignac, Compt. rend., **66**, 181 (1868) *29*

CbH

O_2 **III-831**

When columbium hydride is heated in contact with air, it is converted promptly into columbium pentoxide.

$$2CbH + 3O_2 \rightarrow Cb_2O_5 + H_2O$$

Marignac, Compt. rend., **66**, 181 (1868) *29*

$CbOBr_3$

C_9H_7N

C_5H_5N **III-832**

HBr

10 Milliliters columbium oxybromide solution is heated with 10 ml. 82% hydrogen bromide solution for a short length of time, then a heated solution of 0.12 gm. of quinoline in 6 ml. 25% hydrogen bromide solution is added. On cooling, orange red prisms are formed. The same type reaction occurs with pyridine.

(a) $CbOBr_3 + C_9H_7N + HBr \rightarrow \underline{CbOBr_3 \cdot C_9H_7N \cdot HBr}$

(b) $CbOBr_3 + C_5H_5N + HBr \rightarrow \underline{CbOBr_3 \cdot C_5H_5N \cdot HBr}$

R. F. Weinland and L. Storz, Z. anorg. Chem., **54**, 223 (1907) *28*

$CbOBr_3$

CsBr

RbBr **III-833**

To a heated solution of columbium oxybromide, a heated solution of cesium bromide is added. On cooling, brick red octahedral crystals are obtained. The same type reaction occurs with rubidium bromide.

(a) $CbOBr_3 + 2CsBr \rightarrow \underline{CbOBr_3 \cdot (CsBr)_2}$

(b) $CbOBr_3 + 2RbBr \rightarrow \underline{CbOBr_3 \cdot (RbBr)_2}$

R. F. Weinland and L. Storz, Z. anorg. Chem., **54**, 223 (1907) *28*

$CbOCl_3$

$C_5H_5N \cdot HCl$ **III-834**

A solution of columbium oxychloride is heated to boiling and a warm solution of pyridine hydrochloride is added. On cooling, colorless needles will be obtained. From an alcoholic solution (b) a similar compound will be formed.

(a) $CbOCl_3 + C_5H_5N \cdot HCl \rightarrow CbOCl_3 \cdot C_5H_5N \cdot HCl$

(b) $CbOCl_3 + C_5H_5N \cdot HCl + H_2O \rightarrow CbOCl_3 \cdot C_5H_5N \cdot HCl \cdot H_2O$

R. F. Weinland and L. Storz, Z. anorg. Chem., **54**, 223 (1907) *28*

$CbOCl_3$

$C_9H_7N \cdot HCl$ **III-835**

A solution of columbium oxychloride is heated to boiling and a warm solution of quinoline hydrochloride in concentrated hydrochloric acid is added. On cooling, colorless needles will be obtained. From an alcoholic solution (b) a similar compound will be formed.

(a) $CbOCl_3 + C_9H_7N \cdot HCl \rightarrow \underline{CbOCl_3 \cdot C_9H_7N \cdot HCl}$

(b) $CbOCl_3 + 2(C_9H_7N \cdot HCl) \rightarrow \underline{CbOCl_3 \cdot (C_9H_7N)_2 \cdot 2(HCl)}$

R. F. Weinland and L. Storz, Z. anorg. Chem., **54**, 223 (1907) *28*

$CbOCl_3$

CsCl **III-836**

RbCl

To a solution of columbium oxychloride, which is strongly acidified with hydrochloric acid, a concentrated solution of cesium chloride is added. Octahedral crystals will result. The same type reaction occurs with rubidium chloride.

(a) $CbOCl_3 + 2CsCl \rightarrow CbOCl_3 \cdot (CsCl)_2$

(b) $CbOCl_3 + 2RbCl \rightarrow CbOCl_3 \cdot (RbCl)_2$

R. F. Weinland and L. Storz, Z. anorg. Chem., **54**, 223 (1907) *28*

$CbOCl_3$

Mg **III-837**

By passing the vapor of columbium oxychloride over pure magnesium filaments in a glass tube, magnesium chloride and columbium dioxide are formed.

$$2CbOCl_3 + 3Mg \rightarrow 3MgCl_2 + CbO_2 + (Cb)$$

H. Sainte-Claire Deville and L. Troost, Compt. rend., **60**, 1223 (1865) *29*

$CbOF_3$

NH_4F **III-838**

When a solution containing columbium oxyfluoride is treated with ammonium fluoride and evaporated, a mixed crystalline salt results.

(a) $(3CbOF_3 + NH_4F \rightarrow (CbOF_3)_3 \cdot NH_4F)$

(b) $(3CbOF_3 + 5NH_4F + H_2O \rightarrow (CbOF_3)_3 \cdot (NH_4F)_5 \cdot H_2O)$

C. Marignac, Ann., **4**, 312 (1864) *20*

$CbOF_3 \cdot 2KF$

HF **III-839**

Dissolve $CbOF_3 \cdot 2KF$ in concentrated hydrofluoric acid and cool the solution. Crystals of a double salt separate out.

$$(CbOF_3 \cdot 2KF + 2HF \rightarrow CbF_5 \cdot 2KF + H_2O)$$

C. Marignac, Ann., **4**, sup. 306 (1864) *20*

$CbOF_3 \cdot 2KF$

KF **III-840**

HF

Treat a solution of columbium potassium oxyfluoride with hydrofluoric acid, in which the potassium fluoride is in excess. Concentrate the solution, cool, and collect the crystals. These have the composition as shown:

$$CbOF_3 \cdot 2KF + KF + HF \rightarrow CbOF_3(KF)_3 \cdot HF$$

C. Marignac, Ann., **4**, sup. 302 (1864) *20*

$CbOF_3 \cdot 2KF$

K_2CO_3 **III-841**

A solution of the double salt potassium columbium oxyfluoride is heated with twice its weight of potassium carbonate. A precipitate is formed.

$$6CbOF_3 \cdot 2KF + 10K_2CO_3 + 5H_2O \rightarrow \underline{(Cb_2O_5)_3 \cdot K_2O \cdot 5H_2O} + 20KF + 10CO_2$$

C. Marignac, Ann., **4**, sup. 295 (1864) *20*

CbO_2OH

H_2O_2 **III-842**

Percolumbic acid is obtained by heating columbium hydroxide with a 30% solution of hydrogen peroxide.

$$CbO_2OH + H_2O_2 \rightarrow HCbO_4 + H_2O$$

P. Melikoff and L. Pissarjewsky, Z. anorg. Chem., **20**, 340 (1899)
Ref., C. W. Balke and E. F. Smith, J. Am. Chem. Soc., **30**, 1656 (1908) *1*

CbO_2OH

NaOH **III-843**

Sodium columbate is formed when columbium hydroxide is fused with sodium hydroxide.

$$2CbO_2OH + 2NaOH + 4H_2O \rightarrow Na_2O \cdot Cb_2O_5 \cdot 6H_2O$$

C. W. Balke and E. F. Smith, J. Am. Chem. Soc., **30**, 1647 (1908) *25*

Cb_2O_4

H_2O **III-844**

The heat of formation of columbium tetroxide can be calculated from the following equilibrium:

$$Cb_2O_4 + H_2O \rightleftharpoons Cb_2O_5 + H_2 + 18.2 \text{ cal.}$$

P. Süe, J. chim. phys., **36**, 280 (1939) *69*

Cb_2O_5

C **III-845**

The reduction of columbium pentoxide by carbon takes place at 700–800°C, after two reactions:

(a) $$Cb_2O_5 + C \rightarrow Cb_2O_4 + CO$$

(b) $$Cb_2O_5 + CO \rightarrow Cb_2O_4 + CO_2$$

P. Süe, J. chim. phys., **36**, 280 (1939) *69*

Cb_2O_5

CCl_4 **III-846**

Columbium oxychloride is the main product formed when vapors of carbon tetrachloride are passed over columbium pentoxide heated in a glass tube in an atmosphere of chlorine.

$$3CCl_4 + Cb_2O_5 \rightarrow 2CbOCl_3 + 3COCl_2$$

M. De LaFontaine and C. E. Linebarger, J. Am. Chem. Soc., **18**, 534 (1896) *1*

Cb_2O_5

CCl_4 **III-847**

Columbium oxide is converted into the chloride when the vapors of carbon tetrachloride are passed over it heated below redness.

$$2Cb_2O_5 + 5CCl_4 \rightarrow 4CbCl_5 + 5CO_2$$

C. DeMarcay, Compt. rend., **104**, 111 (1887)
Ref., M. DeLafontaine and C. E. Linebarger, J. Am. Chem. Soc., **18**, 532 (1896) *1*

Cb_2O_5

CaF_2 **III-848**

Calcium fluoride and columbic acid heated to light redness in a stream of hydrogen chloride yields columbic oxyfluoride.

$$3CaF_2 + Cb_2O_5 + 6HCl \rightarrow 2CbOF_3 + (3CaCl_2 + 3H_2O)$$

H. Henninger, Ber. (I), **9**, 191 (1876) *26*

Cb_2O_5

$CbCl_5$ **III-849**

Columbium oxide is placed in a platinum boat and the latter is put in a tube (covered with foil), through which a current of dry CO_2 is passed. An oxychloride is formed by distilling columbium pentachloride over the columbium oxide, which has been heated red hot.

$$Cb_2O_5 + 3CbCl_5 \rightarrow 5CbOCl_3$$

H. Sainte-Claire Deville and L. Troost, Compt. rend., **64**, 294 (1867) *29*

Cb_2O_5

CsOH **III-850**

H_2F_2

A double fluoride of cesium and columbium is formed when solutions of cesium hydroxide in hydrofluoric acid and columbium oxide in hydrofluoric acid are mixed.

$$Cb_2O_5 + 14CsOH + 12H_2F_2 \rightarrow 2(7CsF \cdot CbF_5) + 19H_2O$$

Mary E. Pennington, J. Am. Chem. Soc., **18**, 60 (1896) *1*

Cb_2O_5

H_2 **III-851**

Columbium pentoxide can be reduced to the metal by hydrogen under pressure of 5 atm. and at a temperature of 2500°C.

$$Cb_2O_5 + 5H_2 \rightarrow 2Cb + 5H_2O$$

H. v. Wartenberg, J. Broy, and R. Reinicke, Z. Elektrochem., **29**, 217 (1923) *86*

Cb_2O_5

H_2F_2 **III-852**

Columbium pentoxide is completely changed to fluoride at low red heat by hydrogen fluoride.

$$Cb_2O_5 + 5H_2F_2 \rightarrow 2CbF_5 + (5H_2O)$$

W. K. Van Haagen and E. F. Smith, J. Am. Chem. Soc., **33**, 1504 (1911) *1*

Cb_2O_5

H_2F_2 **III-853**

K_2F_2

Potassium columbium oxyfluoride is formed when columbium oxide is treated with hydrofluoric acid and potassium fluoride.

(a) $$Cb_2O_5 + 3H_2F_2 \rightarrow 2CbOF_3 + 3H_2O$$

(b) $$CbOF_3 + K_2F_2 \rightarrow K_2CbOF_5$$

R. D. Hall, J. Am. Chem. Soc., **26**, 1237 (1904) *1*

Cb_2O_5

KOH **III-854**

When columbium pentoxide (or the hydrated oxide) is fused with potassium hydroxide, potassium columbate is formed.

(a) $Cb_2O_5 + 2KOH \rightarrow (2KCbO_3 + H_2O)$

(b) $(2Cb(OH)_3 + 2KOH + O_2 \rightarrow 2KCbO_3 + 4H_2O)$

A. Atterberg, K. Sven. Vet. Ak. Handl. Övers., **32**, No. 7, 24 (1875) *10*

Cb_2O_5

KOH **III-855**

H_2O_2

Fuse columbium pentoxide with potassium hydroxide. Dissolve in very little water, add hydrogen peroxide and alcohol, a white colored precipitate of potassium percolumbate is obtained.

$$Cb_2O_5 + 4KOH + 4H_2O_2 \rightarrow \underline{K_4Cb_2O_{11}\cdot 3H_2O} + 3H_2O$$

P. Melikoff and L. Pissarjewski, Z. anorg. Chem., **20**, 340 (1899) *28*

Cb_2O_5

K_2CO_3 **III-856**

Potassium columbate is obtained by fusing columbic oxide with potassium carbonate and allowing the melt to crystallize from a water solution.

$$3Cb_2O_5 + 4K_2CO_3 + 16H_2O \rightarrow (K_2O)_4\cdot(Cb_2O_5)_3\cdot 16H_2O + 4CO_2$$

C. Marignac, J. f. prakt. Chem., **97**, 449 (1866)
Ref., C. W. Balke and E. F. Smith, J. Am. Chem. Soc., **30**, 1648 (1908) *1*

Cb_2O_5

K_2CO_3 **III-857**

Columbic acid is fused with 2 to 3 times its weight of potassium carbonate, dissolved in water and concentrated. Crystals of the potassium salt separate.

$$3Cb_2O_5 + 4K_2CO_3 \rightarrow (Cb_2O_5)_3\cdot 4K_2O + 4CO_2$$

C. Marignac, Ann., **3**, sup. 295 (1864) *20*

Cb_2O_5

K_2CO_3 **III-858**

$H_2C_2O_4$

10 Grams of columbium oxide are fused together with 15 grams of potassium carbonate, dissolved in water, then a solution of 28 grams of oxalic acid are added. Evaporate to a volume of about 200 cc. By cooling, crystals of columbium potassium oxalate are obtained. The same type reaction occurs with sodium carbonate and rubidium carbonate.

(a) $Cb_2O_5 + 3K_2CO_3 + 6H_2C_2O_4$
$\rightarrow \underline{Cb_2O_5 \cdot (K_2O)_3 \cdot 6(C_2O_3) \cdot 4H_2O} + 3CO_2 + 2H_2O$

(b) $Cb_2O_5 + 3Na_2CO_3 + 6H_2C_2O_4$
$\rightarrow \underline{Cb_2O_5 \cdot (Na_2O)_3 \cdot 6C_2O_3 \cdot 6H_2O} + 3CO_2$

(c) $Cb_2O_5 + 3Rb_2CO_3 + 6H_2C_2O_4$
$\rightarrow \underline{Cb_2O_5 \cdot Rb_2O_3 \cdot 6C_2O_3 \cdot 4H_2O + 2H_2O} + 3CO_2$

F. Russ, Z. anorg. Chem., **31**, 42 (1902) *28*

Cb_2O_5

K_2CO_3 **III-859**

$NH_4HC_2O_4$

Columbium oxide is fused together with potassium carbonate. The resulting potassium columbate is decomposed with *N*-hydrochloric acid, yielding hydrated columbium oxide. This will react with a solution of ammonium hydrogen oxalate yielding crystals of ammonium columbium oxalate.

(a) $Cb_2O_5 + K_2CO_3 \rightarrow \underline{2KCbO_3} + CO_2$

(b) $2KCbO_3 + 2HCl \rightarrow Cb_2O_5 \cdot H_2O + 2KCl$

(c) $Cb_2O_5 \cdot H_2O + 6NH_4HC_2O_4$
$\rightarrow \underline{Cb_2O_5 \cdot 3(NH_4)_2O \cdot 6C_2O_3 \cdot 3H_2O} + H_2O$

F. Russ, Z. anorg. Chem., **31**, 42 (1902) *28*

Cb_2O_5

NaOH **III-860**

Sodium columbate is formed when columbic oxide is fused with sodium hydroxide in a silver crucible.

$$Cb_2O_5 + 2NaOH + 5H_2O \rightarrow Na_2O \cdot Cb_2O_5 \cdot 6H_2O$$

C. W. Balke and E. F. Smith, J. Am. Chem. Soc., **30**, 1647 (1908) *1*

Cb_2O_5

PCl_5 **III-861**

Columbium oxychloride is formed when columbic oxide is heated in a sealed tube at 180°–200° for seven hours with phosphorus pentachloride and then the tube opened to allow air to enter and heat again applied.

(a) $$Cb_2O_5 + 5PCl_5 \rightarrow 2CbCl_5 + 5POCl_3$$

(b) $$CbCl_5 + O \rightarrow CbOCl_3 + Cl_2$$

Mary E. Pennington, J. Am. Chem. Soc., **18**, 62 (1896) *1*

Cb_2O_5

RbF **III-862**

A double fluoride of columbium and rubidium is formed when columbic oxide is dissolved in hydrofluoric acid and the calculated amount of rubidium fluoride is added.

$$Cb_2O_5 + 5H_2F_2 + 4RbF \rightarrow 2(RbF)_2CbF_5 + 5H_2O$$

Mary E. Pennington, J. Am. Soc., **18**, 58 (1896) *1*

Cb_2O_5

S_2Cl_2 **III-863**

Columbium chloride is formed when columbium oxide is ignited in the vapors of sulfur monochloride and the product distilled in an atmosphere of chlorine.

$$2Cb_2O_5 + 10S_2Cl_2 \rightarrow 4CbCl_5 + 5SO_2 + 15S$$

E. F. Smith and R. Hall, J. Am. Chem. Soc., **26**, 1235 (1904)
Ref., C. W. Balke and E. F. Smith, ibid., **30**, 1640 (1908) *1*

$Cb_2O_5 \cdot H_2O$

H_2O_2 **III-864**

By heating the hydrate of columbium pentoxide with a solution of hydrogen peroxide a yellow colored solution of percolumbic acid is obtained.

$$Cb_2O_5 \cdot H_2O + 2H_2O_2 \rightarrow 2HCbO_4 + 2H_2O$$

P. Melikoff and L. Pissarjewski, Z. anorg. Chem., **20**, 340 (1899) *28*

$Fe(CbO_3)_2$

H_2F_2 **III-865**

Powdered columbite is completely decomposed when exposed to action of hydrogen fluoride gas.

$$Fe(CbO_3)_2 + H_2F_2 \rightarrow Cb_2O_5 + (FeF_2 + H_2O)$$

W. K. Van Haagen and E. F. Smith, J. Am. Chem. Soc., **33**, 1505 (1911) *1*

$Fe(CbO_3)_2$

$KHSO_4$ **III-866**

Finely powdered columbite is converted into columbium tetroxide when fused with at least nine times its weight of potassium acid sulfate for five hours.

$$4Fe(CbO_3)_2 + 12KHSO_4 \rightarrow 4Cb_2O_4 + 6K_2SO_4 + 2Fe_2(SO_4)_3 + 6H_2O + O_2$$

Mary E. Pennington, J. Am. Chem. Soc., **18**, 40 (1896) *1*

$HCbO_3$

HF **III-867**

Columbic acid hydrate dissolved in hydrofluoric acid concentrated and cooled gives crystals of columbium penta fluoride.

$$2HCbO_3 + 10HF \rightarrow (CbF_5)_2 \cdot 5H_2O + H_2O$$

C. Marignac, Ann., **4**, sup. 296 (1864) *20*

$HCbO_3$

KF **III-868**

Dissolve columbic acid in hydrofluoric acid and add a small quantity of potassium fluoride. Concentrate the solution and remove the first crop of crystals. Reconcentrate the mother liquor, cool and remove the second crop of crystals. These correspond to the formula shown below.

$$(3HCbO_3 + 9HF + 5KF \rightarrow (CbOF_3)_3(KF)_5 \cdot H_2O + 5H_2O)$$

C. Marignac, Ann., **4**, sup. 304 (1864) *20*

$HCbO_3$

NH_4F **III-869**

Solution of columbic acid in hydrogen fluoride treated with ammonium fluoride, concentrated and cooled gives crystals of a double salt.

$$(HCbO_3 + 3HF + 2NH_4F \rightarrow CbOF_3 \cdot 2NH_4F + 2H_2O)$$

C. Marignac, Ann., **4**, sup. 308 (1864) *20*

$KCbO_3$

$CuSO_4$ **III-870**

A solution of potassium columbate will react with a solution of copper sulfate yielding a precipitate. This precipitate fused with boric acid yields black crystals.

$$2KCbO_3 + CuSO_4 \rightarrow \underline{CuO \cdot Cb_2O_5} + K_2SO_4$$

A. Larsson, Z. anorg. Chem., **12**, 189 (1896) *28*

$KCbO_3$

$MgCl_2$ **III-871**

Solutions of potassium columbate and magnesium chloride are mixed, the precipitate fused with anhydrous magnesium chloride, extracted with water, treated with hydrochloric acid, and evaporated. There results small tablets and long prisms, perhaps basic magnesium columbate, probably normal magnesium columbate.

(a) $$(2KCbO_3 + MgCl_2 \rightarrow MgO \cdot Cb_2O_5 + 2KCl)$$

(b) $$(MgCl_2 + H_2O \rightarrow MgO + 2HCl)$$

(c) $$(3MgO + Mg(CbO_3)_2) \rightarrow (MgO)_3 \cdot Mg(CbO_3)_2$$

(d) $$(MgO)_3 \cdot Mg(CbO_3)_2 + 6HCl \rightarrow (Mg(CbO_3)_2 + 3MgCl_2 + 3H_2O)$$

A. Larsson, K. Sven. Vet. Ak. Handl. Övers., **52**, No. 9, 706 (1895) *10*

$KCbO_3$

$MgCl_2$ **III-872**

To a solution of potassium columbate is added a solution of magnesium chloride. The resulting precipitate is fused with magnesium chloride yielding a white precipitate. The same type reaction occurs with calcium chloride.

(a) $3KCbO_3 + 4MgCl_2 \rightarrow \underline{(MgO)_4 \cdot Cb_2O_5} + (CbCl_5 + 3KCl)$

(b) $3KCbO_3 + 3CaCl_2 \rightarrow \underline{(CaO)_3 \cdot Cb_2O_5} + (CbOCl_3 + 3KCl)$

A. Larsson, Z. anorg. Chem., **12**, 189 (1896) *28*

$KCbO_3$

$ZnCl_2$ **III-873**

A solution of potassium columbate will react with a solution of zinc chloride. The resulting precipitate is fused with boric acid yielding brown colored crystals of zinc columbate. The same type reaction occurs with cadmium nitrate and cobalt nitrate.

(a) $2KCbO_3 + ZnCl_2 \rightarrow \underline{ZnO \cdot Cb_2O_5} + 2KCl$

(b) $2KCbO_3 + Cd(NO_3)_2 \rightarrow \underline{CdO \cdot Cb_2O_5} + 2KNO_3$

(c) $2KCbO_3 + Co(NO_3)_2 \rightarrow \underline{CoO \cdot Cb_2O_5} + 2KNO_3$

A. Larsson, Z. anorg. Chem., **12**, 189 (1896) *28*

$KCbO_4$

H_2SO_4 **III-874**

Free percolumbic acid is obtained as a yellow amorphous powder when dilute sulfuric acid is added to potassium percolumbate and the clear yellow solution evaporated to dryness.

$$2KCbO_4 + H_2SO_4 \rightarrow \underline{2HCbO_4} + K_2SO_4$$

C. W. Balke and E. F. Smith, J. Am. Chem. Soc., **30**, 1656 (1908) *1*

K_2CbF_7

Al **III-875**

Reduction of potassium fluocolumbate by aluminum in a graphite crucible gives an aluminum columbide, which is obtained as a residue on treating with HCl the residue of aluminum in which it is disseminated.

$$(2K_2CbF_7 + 6Al \rightarrow 2CbAl_3 + 4KF + 5F_2)$$

C. Marignac, Compt. rend., **66**, 181 (1868) *29*

K_2CbF_7

Na **III-876**

Potassium fluocolumbate is reduced without difficulty by sodium in an iron crucible; sodium columbide is formed and is dispersed in the melted mass in the form of a black powder.

$$(2K_2CbF_7 + 2Na \rightarrow 2NaCb + 4KF + 5F_2)$$

C. Marignac, Compt. rend., **66**, 180 (1868)
Ref., Kruss and Nilson., Ber., **20**, 1691 (1887) *29*

K_2CbOF_5

H_2O_2 **III-877**

Crystals of potassium fluoxy percolumbate are formed when potassium columbium fluoride reacts with hydrogen peroxide.

$$K_2CbOF_5 + H_2O_2 \rightarrow \underline{K_2CbO_2F_5 \cdot H_2O}$$

A. Piccini, Z. anorg. Chem., **2**, 21 (1892) *28*

$K_8Cb_6O_{19}$

K_2CO_3 **III-878**

When potassium columbate mixed with a small excess of potassium carbonate is heated to 1200° in a graphite crucible, one obtains a melt in the midst of which black cubes separate. The material which envelopes them can be separated by treatment with HF. Chlorine attacks the crystals and gives a small residue of columbium pentoxide and a mixture of columbium chloride and oxychloride. It is necessary to surround the crucible with a mixture of rutile and carbon. When this is not done, the graphite crucible reduces the K_2CO_3 and the columbium pentoxide, and one obtains a nitride carbide of columbium.

(a) $K_8Cb_6O_{19} + 2K_2CO_3 \rightarrow 3Cb_2O_2 + (6K_2O + 2CO + 11(O))$

(b) $3Cb_2O_2 + 6Cl_2 \rightarrow CbOCl_3 + 3CbCl_3 + Cb_2O_5$

H. St. Claire Deville., Compt. rend., **66**, 183 (1868) *29*

$(MgO)_3 \cdot Mg(CbO_3)_2$

H_3BO_3 **III-879**

Precipitated magnesium columbate is fused with boric acid and the melt extracted with water. The solution yields small, short, prismatic crystals, probably normal magnesium columbate. Similar methods yield corresponding salts of Ca, Zn, Cu, Cd, Co, Y, Mn, Th, and Zr.

$$((MgO)_3 \cdot Mg(CbO_3)_2 + 2H_3BO_3 \rightarrow Mg(CbO_3)_2 + Mg_3(BO_3)_2 + 3H_2O)$$

A. Larsson, K. Sven. Vet. Ak. Handl. Övers., **52**, No. 9, 707–715 (1895) *10*

NaCb

H_2O **III-880**

Sodium columbide is decomposed by water with a feeble evolution of hydrogen and the formation of columbium hydride which is contaminated with some oxygen compounds of columbium.

$$[3NaCb + 8H_2O \rightarrow CbH + 3NaOH + Cb_2O_5 + 6H_2]$$

C. Marignac, Compt. rend., **66**, 181 (1868) *29*

Cu

$AgNO_3$ **III-881**

Silver is precipitated by action of copper metal on silver nitrate solution.

$$Cu + 2AgNO_3 \rightarrow \underline{2Ag} + Cu(NO_3)_2$$

H. V. A. Briscoe and H. F. V. Little, J. Chem. Soc. (London), **105**, 1318 (1914) *57*

Cu

Ag_2SO_4 **III-882**

Copper is separated from other metals in slimes by action of steam and silver sulfate solution.

$$Cu + Ag_2SO_4 \rightarrow CuSO_4 + 2Ag$$

C. Whitehead, J. Am. Chem. Soc., **17**, 851 (1895) *1*

Cu

Br **III-883**

NH_4Br

To finely divided copper, in double the weight of water in a stoppered bottle, add slowly, under cooling, a slight excess of bromine. Now warm in an open dish to expel this excess and add to the resulting solution of cupric bromide as much ammonium bromide as theoretically necessary. Filter the dark wine red solution and let slowly crystallize. Green crystals are obtained.

(a) $$Cu + 2Br \rightarrow CuBr_2$$

(b) $$CuBr_2 + 2NH_4Br + 2H_2O \rightarrow CuBr_2 \cdot 2NH_4Br \cdot 2H_2O$$

L. L. de Koninck, Z. angew. Chem., **1**, 507 (1888) *34*

Cu

$CHCl_3$ **III-884**

Chloroform reacts with metals in the presence of moisture.

$$4CHCl_3 + 2Cu \rightarrow C_2Cl_6 + 2CH_2Cl_2 + Cu_2Cl_2$$

C. Baskerville and W. A. Hamor, J. Ind. Eng. Chem., **4**, 369 (1912) *22*

Cu

CS_2 **III-885**

CO_2

A mixture of carbon dioxide and disulfide vapor passed over copper at high red heat yields carbon monoxide.

$$CS_2 + CO_2 + 4Cu \rightarrow 2CO + 2Cu_2S$$

A. Eiloart, Chem. News, **52**, 184 (1885)
Ref., A. A. B., J. Am. Chem. Soc., **7**, 286 (1885) *1*

Cu

C_2H_2 **III-886**

Copper reacts with acetylene to form a copper acetylide which reacts with more acetylene to form cuprene.

(a) $$nC_2H_2 + nCu \rightarrow nC_2Cu + nH_2$$

(b) $$nC_2Cu + 6nC_2H_2 \rightarrow (C_7H_8)_2n + nCu$$

P. Sabatier and J. B. Senderens, Compt. rend., **130**, 250 (1900)
Ref., J. M. Calhoun, Can. J. Res., **15B**, 221 (1937) *94*

Cu

Cl_2 **III-887**

In testing oils or fats for the presence of chlorinated compds., or halogens in general, a small copper spiral, free from halogens, is dipped in the oil to be tested, ignited and the oil allowed to burn off outside the flame. Upon placing the wire in a non-luminous flame, a green flash indicates the presence of a halogen.

$$Cu + 2Cl \rightarrow CuCl_2$$

F. Beilstein, Ber., **5**, 620 (1872)
Ref., W. L. Ruigh, Anal. Ed., **11**, 250 (1939)
Ref., M. F. Lauro, Oil and Soap, **13**, 2, 45 (1936) *111*

Cu

$CuCl_2$ **III-888**

Precipitated copper reacts with excess cupric chloride solution to form cuprous chloride.

$$Cu + CuCl_2 \rightarrow 2CuCl$$

John P. Riott, Anal. Ed., **13**, 546 (1941) *44*
Ref., G. D. Bengough and R. May, Can. Chem. & Met., **8**, 267 (1924) *15*

Cu

$CuCl_2$ **III-889**

O_2

Action of copper in the presence of oxygen on cupric chloride produces cupric oxychloride.

$$4Cu + 4CuCl_2 + 10H_2O + 2O_2 \rightarrow 2Cu_4(OH)_6Cl_2 \cdot H_2O + (4HCl)$$

G. D. Bengough and R. May, Can. Chem. & Met., **8**, 267 (1924) *15*

Cu

$Cu(OH)_2$ **III-890**

Cominuted copper, in tailings, is oxidized by cupric hydroxide to cuprous oxide, which is soluble in ammonia, when a small amount of ammonium nitrate, or any ammonium salt is added to the solution.

$$(Cu + Cu(OH)_2 \rightarrow (Cu_2O + H_2O)$$

E. A. LeSeur, U.S.P. 755302 (1899)
Ref., L. E. Westman, Can. Chem. & Met., **5**, 29 (1922) *15*

Cu

$FeCl_3$ **III-891**

Metallic copper is attacked by a solution of ferric chloride according to (a). Reaction (b) takes place in solution:

(a) $$Cu + FeCl_3 \rightarrow CuCl + FeCl_2$$

(b) $$CuCl + FeCl_3 \rightarrow CuCl_2 + FeCl_2$$

E. Bekier and S. Trzeciak, J. Chim. phys., **23**, 242 (1926) *69*

Cu

FeS **III-892**

Copper dissolves in fused ferrous sulfide forming cuprous sulfide and iron. Copper behaves similarly with nickel sulfide.

(a) $$2Cu + FeS \rightarrow Cu_2S + Fe$$

(b) $$2Cu + NiS \rightarrow Cu_2S + Ni$$

J. H. L. Vogt, Vid. Sel. Ch. Skrifter., **1**, 26 (1918) *10*

Cu

Fe_2Cl_6 **III-893**

Copper was dissolved in ferric chloride solution, forming cupric chloride; ammonium chloride had to be used to dissolve the copper.

$$Cu + 2FeCl_3 \rightarrow (CuCl_2 + 2FeCl_2)$$

T. J. Baker, Trans. Roy. Soc. (London), **196**, 535 (1901) *105*

Cu

$Fe_2(SO_4)_3$ **III-894**

Ferric sulfate solution dissolves copper to form copper sulfate and ferrous sulfate.

$$Cu + Fe_2(SO_4)_3 \rightarrow CuSO_4 + 2FeSO_4$$

N. S. Keith, Trans. Am. El. Chem. Soc., **1**, 137 (1902) *8*

Cu

H_2SO_4 **III-895**

Pure concentrated sulfuric acid free from arsenic reacts with electrolytic copper at 55°C if held in contact for six hours. The residue contains 8.5% S and 69.7% Cu with oxygen and hydrogen constituting the remainder.

$$Cu + 2H_2SO_4 \rightarrow CuSO_4 + SO_2 + 2H_2O$$

F. de Chaves, Anales Soc. Espan. fis. quim., **29**, 177 (1931) *25*

Cu

HI **III-896**

Metallic copper dissolves slowly in liquid hydriodic acid in a closed tube yielding cuprous iodide.

$$2Cu + 2HI \rightarrow 2CuI + (H_2)$$

R. S. Norris and F. G. Cottrell, Am. Chem. J., **18**, 99 (1896) *1*

Cu

HNO_3 **III-897**

Copper reacts with nitric acid in concentrated copper nitrate solution to form very pure nitrogen.

$$3Cu + 8HNO_3 \rightarrow N_2 + (3Cu(NO_3)_2 + O_2 + 4H_2O)$$

R. Grsstl, Ber., **8**, [1], 831 (1875) *26*

Cu

HNO_3 **III-898**

Copper nitrate can be prepared by treating metallic copper with dilute nitric acid.

$$3Cu + 8HNO_3 \rightarrow 3Cu(NO_3)_2 + 4H_2O + 2NO$$

E. H. Rennie, A. J. Higgin and W. T. Cooke, J. Chem. Soc. (London), **93**, 1168 (1908) *102*
Ref., R. Gerstl, Ber., **8**, (1), 831 (1875) *26*

Cu

HNO_3 **III-899**

Copper is oxidized by nitric acid. Cupric oxide is produced at high temperature.

$$3Cu + 2HNO_3 \rightarrow 3CuO + H_2O + 2NO$$

Oeschsner and Coninck, Rev. gen. chim., **16**, 122 (1913)
Ref., T. E. Thorpe, J. Chem. Soc. (London), **105**, 2578 (1914) *57*

Cu

HNO_3 **III-900**

H_2

In order to prepare pure copper, copper was dissolved in nitric acid; the nitrate was heated to form the oxide and the oxide was reduced by hydrogen.

(a) $$3Cu + 8HNO_3 \rightarrow 3Cu(NO_3)_2 + (2NO + 4H_2O)$$

(b) $$2Cu(NO_3)_2 \rightarrow 2CuO + (4NO_2 + O_2)$$

(c) $$CuO + H_2 \rightarrow Cu + (H_2O)$$

T. J. Baker, Trans. Roy. Soc. (London), **196**, 533 (1901) *105*

Cu

III-901

HNO_3

K_2CO_3

Electrolytic copper was dissolved in nitric acid and precipitated by a solution of potassium carbonate. Distilled water and highly purified potassium carbonate were used.

(a) $Cu + 4HNO_3 \rightarrow Cu(NO_3)_2 + 2NO_2 + (2H_2O)$

(b) $Cu(NO_3)_2 + K_2CO_3 \rightarrow \underline{CuO} + 2KNO_3 + CO_2$

C. R. Wright and A. P. Luff, J. Chem. Soc. (London), **33**, 6 (1878) *81*

Cu

III-902

HNO_3

$Zn(C_2H_3O_2)_2$

KI

Free iodine is liberated when the copper nitrate solution formed by dissolving copper in strong nitric acid is treated with a saturated solution of zinc acetate and then potassium iodide. The resulting solution is then titrated with sodium thiosulfate.

(a) $Cu + 4HNO_3 \rightarrow Cu(NO_3)_2 + 2H_2O + 2NO_2$

(b) $Cu(NO_3)_2 + Zn(C_2H_3O_2)_2 \rightarrow Cu(C_2H_3O_2)_2 + Zn(NO_3)_2$

(c) $2Cu(C_2H_3O_2)_2 + 4KI \rightarrow 2CuI + 4KC_2H_3O_2 + 2I$

A. H. Low, J. Am. Chem. Soc., **18**, 458 (1896) *1*

Cu

H_2SO_4

III-903

There are two simultaneous primary reactions between copper and strong sulfuric acid (Sp. G. 1.843) (a), (b), and two secondary reactions, (c), (d). Some action occurs at 19°C or lower. No sulfide appears at 270° and above.

(a) $Cu + 2H_2SO_4 \rightarrow CuSO_4 + SO_2 + 2H_2O$

(b) $5Cu + 4H_2SO_4 \rightarrow \underline{Cu_2S} + 3CuSO_4 + 4H_2O$

(c) $Cu_2S + 2H_2SO_4 \rightarrow CuS + CuSO_4 + SO_2 + 2H_2O$

(d) $CuS + 2H_2SO_4 \rightarrow S + CuSO_4 + SO_2 + 2H_2O$

S. Pickering, J. Chem. Soc. (London), **33**, 112 (1878) *91*

Cu

H_2SO_4 **III-904**

Sulfur dioxide is formed when copper reacts with concentrated sulfuric acid at 270°C.

$$Cu + 2H_2SO_4 \rightarrow CuSO_4 + SO_2 + 2H_2O$$

C. Baskerville, J. Am. Chem. Soc., **17**, 905 (1895) *1*
Ref., C. W. Rogers, J. Chem. Soc. (London), 268 (1926) *65*

Cu

H_2SO_4 **III-905**

Black cuprous sulfide forms when copper reacts with concentrated sulfuric acid at temperatures between 0°–270°C.

$$5Cu + 4H_2SO_4 \rightarrow \underline{Cu_2S} + 3CuSO_4 + 4H_2O$$

O. B. J. Fraser, D. E. Ackerman and J. W. Sands, Ind. Eng. Chem., **19**, 334 (1927) *23*

Cu

H_2SO_4 **III-906**

O_2

An aerated sulfuric acid solution of low concentration reacts with copper at room temperature to give copper sulfate and water.

$$2Cu + 2H_2SO_4 + O_2 \rightarrow 2CuSO_4 + 2H_2O$$

O. B. J. Fraser, D. E. Ackerman and J. W. Sands, Ind. Eng. Chem., **19**, 333 (1927) *23*

Cu

H_2SO_4 **III-907**

Reaction of metallic copper with hot concentrated sulfuric acid takes place in several steps. Reaction (a) takes place especially at low temperature. Equation (f) is the combined equation for the completed reaction.

(a) $$8Cu + 4H_2SO_4 \rightarrow 3Cu_2SO_4 + \underline{Cu_2S} + 4H_2O$$

(b) $$2Cu + 2H_2SO_4 \rightarrow Cu_2SO_4 + 2H_2O + SO_2$$

(c) $$5Cu_2SO_4 + 4H_2SO_4 \rightarrow \underline{Cu_2S} + 8CuSO_4 + 4H_2O$$

(d) $$Cu_2S + 2H_2SO_4 \rightarrow \underline{CuS} + CuSO_4 + 2H_2O + SO_2$$

(e) $$CuS + 4H_2SO_4 \rightarrow CuSO_4 + 4SO_2 + 4H_2O$$

(f) $$Cu + 2H_2SO_4 \rightarrow CuSO_4 + SO_2 + 2H_2O$$

J. T. Cundall, J. Chem. Soc. (London), **105**, 63 (1914) *57*

Cu

H_2SO_4 **III-908**

O_2

Concentrated sulfuric acid is attacked by copper at ordinary temperature in the presence of air without reduction of the acid.

$$2Cu + O_2 + 2H_2SO_4 \rightarrow 2CuSO_4 + 2H_2O$$

L. Andrews, J. Am. Chem. Soc., **18**, 252 (1896) *1*
Ref., J. F. Thompson and R. J. McKay, Ind. Eng. Chem., **15**, 1115 (1923) *23*

Cu

$Hg(CN)_2$ **III-909**

Copper slowly precipitates mercury from a solution of mecuric cyanide in liquid amonia solution with the formation of a mixture of cuprous and cupric cyanides.

$$3Cu + 2Hg(CN)_2 \rightarrow \underline{2Hg} + 2CuCN + Cu(CN)_2$$

F. W. Bergstrom, J. Am. Chem. Soc., **48**, 2326 (1926) *1*

Cu

$Hg(CNO)_2$ **III-910**

A grayish colored precipitate is formed when 20 ml of plastic copper amalgam is shaken for a long time under water in an atmosphere of hydrogen with mercuric fulminate.

$$2Cu + Hg(CNO)_2 \rightarrow \underline{2CuCNO} + Hg$$

Lothar Wöhler and F. Martin, Ber., **50**, 586 (1917) *28*

Cu

Hg_2SO_4 **III-911**

In lead standard cells containing copper sulfate there is a chemical action between copper and Hg_2SO_4

$$Cu + Hg_2SO_4 \rightarrow CuSO_4 + 2Hg$$

M. G. Mellon and W. E. Henderson, J. Am. Chem. Soc., **42**, 687 (1920) *16*

Cu

I_2 **III-912**

By slightly heating copper with iodine, the result will be cuprous iodide and iodine.

$$2Cu + 2I_2 \rightarrow 2CuI + I_2$$

E. Oliveri Mandata, Gazz. chim. ital., **40**, 107 (1910) *21*

Cu

I_2 **III-913**

Metallic copper reacts with iodine in hydriodic acid solution to form cuprous iodide.

$$2Cu + I_2 \rightarrow 2CuI$$

Ed. Meusel, Ber., **3**, 123 (1870) *11*

Cu

$KClO_3$ **III-914**

H_2SO_4

When strips of copper are dipped into a solution of $KClO_3$ containing sulfuric acid, the metal dissolves slowly and potassium chloride (and a little hypochlorite) is formed. The same reaction proceeds with zinc but more slowly.

(a) $3Cu + KClO_3 + 3H_2SO_4 \rightarrow (3CuSO_4) + KCl + 3H_2O$

(b) $2Cu + KClO_3 + 2H_2SO_4 \rightarrow (2CuSO_4) + KClO + 2H_2O$

A. L. Voege, J. Phys. Chem., **3**, 577 (1899) *7*

Cu

Mg **III-915**

When copper and magnesium are fused, two compounds are produced: $CuMg_2$; m.p. 570°, polygonal crystals; and Cu_2Mg, m.p. 797°, rod-shaped crystals.

(a) $2Cu + Mg \rightarrow Cu_2Mg$

(b) $Cu + 2Mg \rightarrow CuMg_2$

R. Sahmen, Z. anorg. Chem., **57**, 1–33 (1908)
Ref., G. Senter, J. Chem. Soc. (London), **94:2**, 186–187 (1908) *57*

Cu

NH_2OH **III-916**

Copper reacts with hydroxylamine and oxygen, and decomposes it to nitrous oxide.

$$2NH_2OH + 4Cu + 2O_2 \rightarrow N_2O + 2Cu_2O + 3H_2O$$

B. Suler, Z. Elektrochem, **7**, p. 836 (1901) *86*

Cu

$(NH_4)HS$ **III-917**

Copper wire treated with ammonium hydrosulfide gives cuprous sulfide.

$$(NH_4)HS + 2Cu \rightarrow Cu_2S + NH_3 + H_2$$

K. Heumann, Ann., **173**, 29 (1874)
Ref., E. Priwoznik, ibid., **171**, 110 (1874) *20*

Cu

NH_4HS **III-918**

Ammonium bisulfide reacts with metallic copper to form cuprous sulfide, hydrogen, and ammonium sulfide.

$$2NH_4HS + 2Cu \rightarrow Cu_2S + (NH_4)_2S + H_2$$

E. Priwoznik, Ber., **6**, 1292 (1873) *11*

Cu

NH_4NO_3 **III-919**

N_2O

HNO_3

A fairly complicated reaction probably not understood occurs when nitrous oxide reacts with copper. Ackworth reports nitrogen and nitric oxide as products.

$$NH_4NO_3 + 4HNO_3 + N_2O + Cu \rightarrow (O_2) + (4H_2O) + N_2 + 4NO + (Cu(NO_3)_2)$$

R. Gerstl, Ber., **8** [1], 831 (1875) *26*

Cu

$(NH_4)_2CO_3$ **III-920**

NH_3

Cupric ammonium carbonate solution was prepared by agitating, with air, a solution of 15% ammonium carbonate and 5% ammonium hydroxide (by weight), in a tower filled with copper turnings until the copper content was about 3.5%.

$$Cu + (NH_4)_2CO_3 + 2NH_3 + \tfrac{1}{2}O_2 \rightarrow Cu(NH_3)_4CO_3 + H_2O$$

W. R. Hainsworth and E. V. Titus, J. Am. Chem. Soc., **42**, 2 (1921) *16*

Cu

$(NH_4)_2S$ **III-921**

Concentrated ammonium sulfide reacts with metallic copper to form cuprous sulfide, hydrogen and ammonia.

$$(NH_4)_2S + 2Cu \rightarrow Cu_2S + 2NH_3 + H_2$$

E. Priwoznik, Ber., **6**, 1292 (1873) *11*

Cu

$(NH_4)_2Sx$ **III-922**

Granulated copper or copper wire immersed in a solution of yellow ammonium sulfide causes evolution of hydrogen and a series of compounds are formed. A compound which analyzes $Cu_2(NH_4)_2S_7$ is present in the liquid and may be separated by crystallization.

(a) $$Cu + (NH_4)_2S_x \rightarrow CuS + 2NH_3 + H_2 + Sx$$

(b) $$CuS + Cu \rightarrow Cu_2S$$

K. Heumann, Ann., **173**, 21 (1874) *20*

Cu

NO_2 **III-923**

A compound, stable in dry air, is produced when powdered copper reacts with nitrogen peroxide at 25°.

$$2Cu + 2NO_2 \rightarrow 2CuNO_2$$

P. Sabatier and J. B. Senderens, Ann. chim. phys. [7], 399 (1896)
Ref., Tartar and Semon, J. Am. Chem. Soc., **43**, 494 (1921) *1*

Cu

NaCl **III-924**

ε

In the electrolysis of sodium chloride solution with copper electrodes, cuprous chloride is formed at the anode and dilute sodium hydroxide at the cathode.

$$2Cu + 2NaCl + 2H_2O \rightarrow Cu_2Cl_2 + 2NaOH + H_2$$

W. D. Bancroft, Trans. Am. El. Chem. Soc., **8**, 47 (1905) *8*

Cu

Na_2SO_4 **III-925**

ε

In the electrolysis of a sodium sulfate solution with copper electrodes, copper sulfate is formed at the anode and caustic soda at the cathode.

$$Cu + Na_2SO_4 + 2H_2O \rightarrow CuSO_4 + 2NaOH + H_2$$

W. D. Bancroft, Trans. Am. El. Chem. Soc., **8**, 46 (1905) *8*

Cu

Cl^- **III-926**

O_2

Copper metal, when immersed in chloride solution, becomes coated with almost insoluble cuprous chloride. This in turn may become oxidized according to the reaction.

(a) $$Cu + Cl^- \rightarrow CuCl$$

(b) $$4CuCl + \tfrac{1}{2}O_2 \rightarrow Cu_2O + 2CuCl_2$$

E. E. Thum, Chem. & Met. Eng., **26**, 301 (1922) *44*

Cu

O_2 **III-927**

Copper is transformed into cupric oxide by passing a current of oxygen over the red hot metal.

$$(2Cu + O_2 \rightarrow 2CuO)$$

W. Clark, Chem. News., **8**, 61 (1863) *101*

Cu

O_2 **III-928**

Δ

In order to prepare nitrogen, air was passed over hot copper.

$$2Cu + O_2 \rightarrow (2CuO)$$

R. J. Strutt, Trans. Roy. Soc. (London), **193**, 385 (1900) *105*

Cu

S **III-929**

Copper turnings or powder react with a solution of sulphur in carbon disulfide to form copper sulfide.

$$Cu + S \rightarrow CuS$$

H. H. Champion and H. Pellet,
Ref., A. Henninger, Ber. [II], **8**, 1470 (1875) *26*

Cu

S **III-930**

Copper and sulfur when melted together form cuprous sulfide.

$$2Cu + S \rightarrow Cu_2S$$

Karl Heumann, Ber., **6**, 749 (1873) *11*

Cu

SO_2 **III-931**

At 730°, sulfur dioxide reacts with copper forming cuprous sulfide and cuprous oxide.

$$6Cu + SO_2 \rightarrow Cu_2S + 2Cu_2O$$

C. M. Stubbs, J. Chem. Soc. (London), **103**, 1445 (1913) *67*

Cu

$S_2O_8^{=}$ **III-932**

Dilute ammonium persulfate solutions (~ 0.1 % in water) cause corrosion of various metals at room temperature. Sulfates are formed and the metals behave as they do when used as anodes in the electrolysis of sulfates. Copper, nickel, cadmium, aluminum and iron are corroded. Iron dissolves partly as ferrous and partly as ferric ions. Aluminum, it is to be noted, is only slightly attacked.

$$Cu + S_2O_8^{=} \rightarrow Cu^{++} + 2SO_4^{=}$$

J. W. Turrentine, J. Phys. Chem., **11**, 623 (1907) *7*

Cu

$SeCl_4$ **III-933**

Heating selenium tetrachloride with freshly reduced copper gauze to 105° for several hours in the absence of air produces copper selenide (b). Excess chloride yields metallic selenium (c).

(a) $3Cu + 2SeCl_4 \rightarrow 3CuCl_2 + Se_2Cl_2$

(b) $5Cu + Se_2Cl_2 \rightarrow 2Cu_2Se + CuCl_2$

(c) $2Cu_2Se + 4Se_2Cl_2 \rightarrow 4CuCl_2 + 10Se$

W. W. Taylor, E. B. R. Prideaux, and H. G. Pool, J. Am. Chem. Soc., **48**, 2130 (1926) *16*

Cu

$SiCl_4$ **III-934**

Silicon tetrachloride passed over copper at 1200°C, transforms the copper into Cu_4Si and the cuprous chloride volatilizes.

$$SiCl_4 + 8Cu \rightarrow Cu_4Si + 2Cu_2Cl_2$$

E. Vigouroux, Procès-verbaux des séances de la Société des Sciences Physiques et Naturelles de Bordeaux (18 July 1901)
Ref., P. Lebeau, Bull. soc. chim. (France), **1**, 110 (1907) *31*

Cu

Sn **III-935**

The phase rule system copper-tin was studied. The phases present were monocupro stannide, tricupro stannide, and tetracupro stannide.

(a) $Cu + Sn \rightarrow CuSn$

(b) $3Cu + Sn \rightarrow Cu_3Sn$

(c) $4Cu + Sn \rightarrow Cu_4Sn$

C. T. Heycock and T. H. Neville, Trans. Roy. Soc. (London), **202**, 9 (1904) *105*

Cu

Sn **III-936**

Only one compound, namely, Cu_3Sn, is formed when liquid copper-tin alloys are cooled. Five series of solid solutions, however, are observed in these systems. The previously claimed Cu_4Sn is not a compound but a solid solution.

(a) $$3Cu + Sn \rightarrow \underline{Cu_3Sn}$$

(b) $$4Cu + Sn \neq \underline{Cu_4Sn}$$

E. S. Shepherd and E. Blough, J. Phys. Chem., **10**, 630 (1906) *7*

Cu

Te **III-937**

Cuprous telluride is formed when copper and tellurium unite.

$$2Cu + Te \rightarrow Cu_2Te$$

Magottet, These de Paris (1879)
Ref., C. A. Tibbals, J. Am. Chem. Soc., **31**, 909 (1909) *1*

Cu

Zn **III-938**

Cooling liquid solutions of metallic copper and zinc leads to several series of solid solutions but not to compounds.
This corrects information found in the literature.

$$Cu + 2Zn \neq CuZn_2$$

E. S. Shepherd, J. Phys. Chem., **8**, 421 (1904) *7*

Cu

Air (O) **III-939**

Thin strips of electrolytic copper were roasted for many days at bright heat in a porcelain tube through which air was aspirated. The oxide was crushed, particles of metal picked out and the process repeated in air and then in oxygen for several hours giving a product analyzing 194 parts cupric and 3 parts cuprous oxide.

(a) $$2Cu + O_2 \rightarrow 2CuO$$

(b) $$4Cu + O_2 \rightarrow 2Cu_2O$$

C. R. Wright and A. P. Luff, J. Chem. Soc. (London), **33**, 7 (1878) *25*

Cu^{++}

Cl^- **III-940**

$CuCl_2$

Changes in color brought about in solutions of copper salts by changes in concentration and temperature as well as by addition of certain halogen salts are to be attributed to the formation of complex anions, as shown below.

$$Cu^{++} + 2Cl^- + xCuCl_2 \rightarrow Cu^{++} + Cl_2 \cdot (CuCl_2)_x^-$$

F. G. Donnan and H. Bassett, J. Chem. Soc. (London), **81**, 939 (1902)
Ref., Kohlschütter, Ber., **37**, 1193 (1904)
Ref., Stewart B. Watkins and Henry G. Denham, J. Chem. Soc. (London), **115**:**2**, 1269 (1919) *48*

Cu^{++}

$CuBr_2$ **III-941**

Br^-

Migration number measurements indicate that in concentrated solutions, copper bromide is forced into a complex anion; this form of complex is an auto-complex.

$$Cu^{++} + 2Br^- + xCuBr_2 \rightarrow Cu^{++} + Br_2(CuBr_2)_x^-$$

Stewart B. Watkins and Henry G. Denham, J. Chem. Soc. (London), **115**:**2**, 1278 (1919) *48*

Cu^{++}

NaOH **III-942**

Sodium hydroxide precipitates basic salts, corresponding closely to a $\frac{1}{3}$ ratio, from solutions of copper sulfate, nitrate and chloride. At low or extremely high pH's the basic nature of these salts varies.

$$4CuSO_4 + 6NaOH + xH_2O \rightarrow CuSO_4 \cdot (Cu(OH)_2)_3 \cdot xH_2O + 3Na_2SO_4$$

A. J. Hopkins and R. A. Beebe, J. Phys. Chem., **34**, 578 (1930) *85*

Cu^{++}

ϵ **III-943**

In a solution of a copper salt, the mechanism of the electrode process may be represented by three equations: (a) cupric ion reduced to cuprous ion; (b) cuprous ion reduced to metallic copper; (c) cupric ion reduced to metallic copper.

(a) $Cu^{++} + \epsilon \rightarrow Cu^{+}$

(b) $Cu^{+} + \epsilon \rightarrow Cu$

(c) $Cu^{++} + 2\epsilon \rightarrow Cu$

Stewart B. Watkins and Henry G. Denham, J. Chem. Soc. (London), **115**:2, 1276 (1919) *48*

CuBr

C_2H_2 **III-944**

KBr

By passing acetylene into a concentrated solution of cuprous bromide in potassium bromide, a reddish precipitate is formed, which becomes dark brown upon washing with a saturated solution of potassium bromide.

(a) $2CuBr + C_2H_2 + KBr \rightarrow (\underline{HC_2Cu_2Br \cdot KBr} + HBr)$

(b) $HC_2Cu_2Br \cdot KBr \rightarrow HC_2Cu_2Br + KBr$

Bertholet, Compt. rend., **62**, 457 (1866) *29*

CuBr

$C_2H_5 \cdot S \cdot CH_2COOH$ **III-945**

HBr

Cuprous bromide dissolves in ethylthioglycolic acid and yields on evaporation colorless prisms or needles of anhydrous ethylthioglycolic acid cuprous bromide. If dilute hydrobromic acid is present the mono-hydrated compound is formed as needle crystals.

(a) $CuBr + 2C_2H_5 \cdot S \cdot CH_2COOH \rightarrow (C_2H_5 \cdot S \cdot CH_2COOH)_2 \cdot CuBr$

(b) $2CuBr + C_2H_5 \cdot S \cdot CH_2COOH + H_2O + HBr$
$\rightarrow C_2H_5 \cdot S \cdot CH_2COOH \cdot (CuBr)_2 \cdot H_2O + HBr$

L. Ramberg, Acta Univ. Lund. Med. Matem. Natur. Nova Ser., **4**, 2, 11 (1908) *10*

CuBr

C_5H_5N **III-946**

Addition of 10 g. copper bromide to an aqueous solution containing 20 cc pyridine precipitates the complex as a green-yellow crystalline solid.

$$2CuBr + 4C_5H_5N \rightarrow \underline{(Cu_2(C_5H_5N)_4)Br_2}$$

D. O. Marouhian, J. Gen. Chem. (USSR), **10**, 918 (1940) *60*

CuBr

NH_3 **III-947**

Dissolve 10 grams of cuprous bromide in a very small amount of ammonia and 25 ml of acetic acid. If performed in a hydrogen atmosphere, colorless crystals are obtained after 1–2 days.

$$2CuBr + 2NH_3 \rightarrow \underline{Cu_2Br_2 \cdot 2NH_3}$$

T. W. Richards and B. S. Merigold, Z. anorg. Chem., **17**, 245 (1898) *28*

$CuBrC_2H_3O_2 \cdot 2NH_3$

NH_3 **III-948**

Cupriammonium acetobromide absorbs three molecules of ammonia when placed in a freezing mixture in a glass tube.

$$CuBrC_2H_3O_2 \cdot 2NH_3 + 3NH_3 \rightarrow CuBrC_2H_3O_2 \cdot 5NH_3$$

Richards and Forsythe, Proc. Am. Acad., **32**, 239
Ref., J. Am. Chem. Soc., **19**, 104 (1897) *1*

$CuBrC_2H_3O_2 \cdot 5NH_3$

(Air) **III-949**

The above compound loses ammonia on exposure to air passing over into a green compound $CuBr_2 \cdot 6NH_3$.

$$2CuBrC_2H_3O_2 \cdot 5NH_3 \rightarrow CuBr_2 \cdot 6NH_3 + Cu(C_2H_3O_2)_2 \cdot 4NH_3$$

Richards and Forsythe, Proc. Am. Acad., **32**, 239
Ref., C. B. Dudley, J. Am. Chem. Soc., **19**, 104 (1897) *1*

$CuBr_2$

NH_4SCN

NH_3

III-950

Bring 10 grams of cupric bromide in contact with 15 ml. concentrated ammonia. Dissolve the precipitate of cupric ammoniate by adding a small amount of acetic acid. Blue crystals will be formed when a concentrated solution of ammonium thiocyanate is added.

$$CuBr_2 + 4NH_3 + 2NH_4SCN \rightarrow \underline{Cu(SCN)_2 \cdot 4(NH_3)} + 2NH_4Br$$

T. W. Richards and B. S. Merigold, Z. anorg. Chem., **17**, 245 (1898) *28*

$CuBr_2$

Δ

III-951

When cupric bromide is heated, it decomposes to yield cuprous bromide and free bromine.

$$2CuBr_2 \rightarrow 2CuBr + Br_2$$

C. G. Jackson, J. Chem. Soc. (London), **99**:1, 1066 (1911) *103*

$CuBr_2 \cdot NH_4Br$

NH_4CNS

NH_4OH

III-952

Diammonium cupriammonium thiocyanate is prepared by dissolving cupriammonium bromide in a little acetic acid and adding to this a saturated solution of ammonium thiocyanate and allowing the precipitate formed to dissolve in ammonium hydroxide.

$$CuBr_2 \cdot NH_4Br + 2NH_4CNS + NH_4OH \rightarrow Cu(CNS)_2 \cdot 4(NH_3) + 3HBr + H_2O$$

Richards and Merigold, Proc. Am. Acad., **33**, 131–38
Ref., J. Am. Chem. Soc., **20**, 39 (1898) *1*

$Cu(CHO_2)_2$

Pb **III-953**

Copper formate gives a deep blue, poorly conducting solution in pyridine. The dilute and slightly warmed solution quickly deposits copper on metallic lead (a). The same type reaction occurs with copper acetate (b).

(a) $Cu(CHO_2)_2 + Pb \rightarrow \underline{Cu} + Pb(CHO_2)_2$

(b) $Cu(C_2H_3O_2)_2 + Pb \rightarrow \underline{Cu} + Pb(C_2H_3O_2)_2$

J. L. Sammis, J. Phys. Chem., **10**, 593 (1906) *7*

$Cu(CH_3COO)_2$

K_2SO_4 **III-954**

Na_2SO_4

$(NH_4)_2SO_4$

$CuSO_4$

When a hot solution of copper acetate reacts with potassium sulfate, a blue-greenish precipitate of basic copper sulfate, insoluble in water is obtained. The same type reaction occurs with sodium sulfate, ammonium sulfate, and copper sulfate.

(a) $4Cu(CH_3COO)_2 + K_2SO_4 + 7H_2O$
$\rightarrow \underline{CuSO_4 \cdot 3CuO \cdot 4H_2O} + 2KC_2H_3O_2 + 6HC_2H_3O_2$

(b) $4Cu(CH_3COO)_2 + Na_2SO_4 + 7H_2O$
$\rightarrow \underline{CuSO_4 \cdot 3CuO \cdot 4H_2O} + 2NaC_2H_3O_2 + 6HC_2H_3O_2$

(c) $4Cu(CH_3COO)_2 + (NH_4)_2SO_4 + 7H_2O$
$\rightarrow \underline{CuSO_4 \cdot 3CuO \cdot 4H_2O} + 2NH_4C_2H_3O_2 + 6HC_2H_3O_2$

(d) $3Cu(CH_3COO)_2 + CuSO_4 + 7H_2O$
$\rightarrow \underline{CuSO_4 \cdot 3CuO \cdot 4H_2O} + 6HC_2H_3O_2$

W. Casselmann, Z. anal. Chem., **4**, 24 (1865) *28*

$Cu(CH_3COO)_2 \cdot H_2O$

Δ **III-955**

Cupric acetate on heating, decomposes to cuprous acetate, water, acetic acid, acetone, copper and other unidentified products.

(a) $2Cu(CH_3COO)_2 \cdot H_2O \rightarrow Cu_2(CH_3COO)_2 + H_2O$

(b) $Cu_2(CH_3COO)_2 \rightarrow (CH_3)_2CO + Cu_2O + CO_2$

A. Angel and A. V. Harcourt, J. Chem. Soc. (London), **91**, 1385 (1907) *13*

CuCN

$Ba(CN)_2$ **III-956**

$Sr(CN)_2$

$Mg(CN)_2$

Crystals of barium cuprous cyanide are formed when a solution of barium cyanide is saturated with cuprous cyanide. The same type reaction occurs with strontium cyanide and magnesium cyanide.

(a) $2CuCN + Ba(CN)_2 + 4H_2O \rightarrow \underline{BaCu_2(CN)_4 \cdot 4H_2O}$

(b) $2CuCN + Sr(CN)_2 + 8H_2O \rightarrow \underline{SrCu_2(CN)_4 \cdot 8H_2O}$

(c) $2CuCN + Mg(CN)_2 + 11H_2O \rightarrow \underline{MgCu_2(CN)_4 \cdot 11H_2O}$

H. Grossmann and P. v. d. Forst, Z. anorg. Chem., **43**, 94 (1905) *28*

CuCN

$Ca(CN)_2$ **III-957**

The reaction of cuprous cyanide with calcium cyanide will yield colorless crystals of calcium cuprous cyanide.

$$CuCN + Ca(CN)_2 + 4H_2O \rightarrow \underline{CaCu(CN)_3 \cdot 4H_2O}$$

H. Grossmann and P. v. d. Forst, Z. anorg. Chem., **43**, 94 (1905) *28*

CuCN

CsCN **III-958**

A solution of cuprous cyanide is treated with a solution of cesium cyanide. Crystals of cesium cuprous cyanide are formed.

$$2CuCN + 2CsCN + 3H_2O \rightarrow \underline{(CsCu(CN)_2)_2 \cdot 3H_2O}$$

H. Grossmann and P. v. d. Forst, Z. anorg. Chem., **43**, 94 (1905) *28*

CuCN

Cs_2CO_3 **III-959**

HCN

CaO

A solution, containing cuprous cyanide, hydrocyanic acid and cesium carbonate is concentrated. By leaving in a vacuum desiccator for about 8 days, crystals of cesium cuprous cyanide are obtained. The same type reaction occurs with calcium oxide.

(a) $3CuCN + 2HCN + Cs_2CO_3 \rightarrow \underline{Cs_2Cu_3(CN)_5} + CO_2 + H_2O$

(b) $3CuCN + 2HCN + CaO \rightarrow CaCu_3(CN)_5 + H_2O$

H. Grossmann and P. v. d. Forst, Z. anorg. Chem., **43**, 94 (1905) *28*

CuCN

KCNS **III-960**

NH_4CNS

Cuprous cyanide is easily soluble in a very concentrated solution of potassium thiocyanate or ammonium thiocyanate. On evaporating, double salts are formed.

(a) $2CuCN + 3KCNS \rightarrow (CuCN)_2 \cdot 3KCNS$

(b) $2CuCN + 3NH_4CNS \rightarrow (CuCN)_2 \cdot 3NH_4CNS$

H. Grossmann, Z. anorg. Chem., **37**, 407 (1903) *28*

CuCN

$K_3Fe(CN)_6$ **III-961**

$Na_3Fe(CN)_6$

By boiling cuprous cyanide with a solution of potassium ferricyanide a precipitate of brown color is obtained. The same type reaction occurs with sodium ferricyanide.

(a) $CuCN + 2K_3Fe(CN)_6 \rightarrow \underline{K_2CuFe(CN)_6} + K_4Fe(CN)_6 + CN$

(b) $CuCN + 2Na_3Fe(CN)_6 \rightarrow \underline{Na_2CuFe(CN)_6 + Na_4Fe(CN)_6} + CN$

J. Messner, Z. anorg. Chem., **9**, 126 (1895) *28*

CuCN

$Li_4Fe(CN)_6$ **III-962**

By boiling cuprous cyanide with lithium ferrocyanide colorless crystals are obtained. The reaction must be carried out in an atmosphere of hydrogen.

$$2CuCN + Li_4Fe(CN)_6 \rightarrow \underline{Li_2Cu_2Fe(CN)_6} + 2LiCN$$

J. Messner, Z. anorg. Chem., **9**, 126 (1895) *28*

CuCN

$(NH_4)_4Fe(CN)_6$ **III-963**

$Mg_2Fe(CN)_6$

By boiling cuprous cyanide with a solution of ammonium ferrocyanide in a stream of hydrogen, colorless crystals of ammonium cuproferrocyanide are obtained. The same type reaction occurs with magnesium ferrocyanide.

(a) $2CuCN + (NH_4)_4Fe(CN)_6 \rightarrow \underline{(NH_4)_2Cu_2Fe(CN)_6} + 2NH_4CN$

(b) $2CuCN + Mg_2Fe(CN)_6 \rightarrow \underline{MgCu_2Fe(CN)_6} + Mg(CN)_2$

J. Messner, Z. anorg. Chem., **8**, 368 (1895) *28*

CuCN

NaCN **III-964**

Colorless crystals of sodium cuprous cyanide will crystallize from a concentrated solution of cuprous cyanide in sodium cyanide.

$$CuCN + 3NaCN + 3H_2O \rightarrow \underline{Na_3Cu(CN)_4 \cdot 3H_2O}$$

H. Grossmann and P. v. d. Forst, Z. anorg. Chem., **43**, 94 (1905) *28*

CuCN

NaCN **III-965**

NH_4CN

LiOH

By saturating a very concentrated solution of sodium cyanide with cuprous cyanide colorless crystals of sodium cuprous cyanide are obtained. The same type reaction occurs with ammonium cyanide and lithium hydroxide.

(a) $CuCN + NaCN + 2H_2O \rightarrow \underline{NaCu(CN)_2 \cdot 2H_2O}$

(b) $2CuCN + NH_4CN + H_2O \rightarrow \underline{NH_4 \cdot Cu_2(CN)_3 \cdot H_2O}$

(c) $CuCN + NH_4CN \rightarrow \underline{NH_4Cu(CN)_2}$

(d) $2CuCN + LiOH + HCN + 2H_2O \rightarrow LiCu_2(CN)_3 \cdot 3H_2O$

H. Grossmann and P. v. d. Forst, Z. anorg. Chem., **43**, 94 (1905) *28*

CuCN

$Na_4Fe(CN)_6$ **III-966**

$K_4Fe(CN)_6$

By boiling cuprous cyanide with a solution of sodium ferrocyanide colorless crystals of sodium cuproferro cyanide are obtained. The same type reaction occurs with potassium ferrocyanide.

(a) $2CuCN + K_4Fe(CN)_6 \rightarrow \underline{K_2Cu_2Fe(CN)_6} + 2KCN$

(b) $2CuCN + Na_4Fe(CN)_6 \rightarrow \underline{Na_2Cu_2Fe(CN)_6} + 2NaCN$

J. Messner, Z. anorg. Chem., **8**, 368 (1895) *28*

CuCN

Rb_2CO_3 **III-967**

HCN

A solution of 12% hydrocyanic acid containing cuprous cyanide and rubidium carbonate is concentrated on the water bath. Crystals of rubidium cuprous cyanide are formed.

$$2CuCN + 2HCN + Rb_2CO_3 \rightarrow \underline{2RbCu(CN)_2} + CO_2 + H_2O$$

H. Grossmann and P. v. d. Forst, Z. anorg. Chem., **43**, 94 (1905) *28*

CuCN

Se **III-968**

A liquid ammonia solution of cuprous cyanide (formed by the action of excess copper in liquid ammonia on mercuric cyanide) reacts slowly with selenium to form a blue solution of cupric selenocyanate and a black precipitate of cupric selenide.

$$2CuCN + 3Se \rightarrow Cu(SeCN)_2 + \underline{CuSe}$$

F. W. Bergstrom, J. Am. Chem. Soc., **48**, 2326 (1926) *16*

CuCN · NaCN

$K_4Fe(CN)_6$ **III-969**

$Na_4Fe(CN)_6$

A solution of cuprous cyanide-sodium cyanide will react with a saturated solution of sodium ferrocyanide yielding colorless crystals of sodium cuprous ferrocyanide. The same type reaction occurs with potassium ferrocyanide.

(a) $2[CuCN \cdot NaCN] + K_4Fe(CN)_6 \rightarrow \underline{Na_2Cu_2Fe(CN)_6} + 4KCN$

(b) $2[CuCN \cdot NaCN] + Na_4Fe(CN)_6 \rightarrow \underline{Na_2Cu_2Fe(CN)_6} + 4NaCN$

J. Messner, Z. anorg. Chem., **8**, 368 (1895) *28*

CuCNS

$KMnO_4$ **III-970**

H_2SO_4

Cuprous thiocyanate is oxidized by potassium permanganate in the presence of sulfuric acid.

$$10CuCNS + 14KMnO_4 + 21H_2SO_4 \rightarrow 7K_2SO_4 + 14MnSO_4 + 10HCN + 10CuSO_4 + 16H_2O$$

S. W. Parr, J. Am. Chem. Soc., **22**, 685 (1900)
Ref., J. Am. Chem. Soc., **23**, 34 (1901) *1*

CuCNS

NaOH **III-971**

Cuprous thiocyanate is decomposed by hot caustic soda forming cuprous hydroxide and sodium thiocyanate.

$$CuCNS + NaOH \rightarrow CuOH + NaCNS$$

L. F. Clark, J. Ind. Eng. Chem., **11**, 1138 (1919) *22*

$Cu(CN)_2$

Δ **III-972**

At a temperature below 100°, cupric cyanide gives off cyanogen and leaves a residue of cuprous cyanide.

$$2Cu(CN)_2 \rightarrow Cu_2(CN)_2 + (CN)_2(\text{"}CyCu_2\text{"})$$

A. Lallemand, Compt. rend., **58**, 750 (1864) *29*

$Cu(CN)_2 \cdot 6KCN$

$AgNO_3$ **III-973**

Silver nitrate added to a solution of copper potassium cyanide breaks off four molecules of potassium cyanide to form the double salt.

$$Cu(CN)_2 \cdot 6KCN + 2AgNO_3 \rightarrow Cu(CN)_2 \cdot 2KCN + 2AgCN \cdot KCN + 2KNO_3$$

A. Brochet and J. Petit, Ann. chim. phys. [VIII], **3**, 444 (1904–05) *100*

$Cu(CNS)_2 \cdot 4NH_3$

NH_3 **III-974**

Copper thiocyanate tetrammoniate will react with ammonia at low temperature yielding copper thiocyanate hexammoniate.

$$Cu(CNS)_2 \cdot 4NH_3 + 2NH_3 \rightarrow \underline{Cu(CNS)_2 \cdot 6NH_3}$$

Fritz Ephraim and Edward Bolle, Ber., **48**, 1770 (1916) *28*

$CuCO_3$

$CH_2ClCOOH$ **III-975**

Copper monochloroacetate forms when copper carbonate is added to a water and absolute alcohol solution of monochloroacetic acid.

$$2CH_2ClCOOH + CuCO_3 + 4H_2O \rightarrow Cu(CH_2ClCOO)_2 \cdot 4H_2O + (H_2CO_3)$$

W. G. Bateman and A. B. Hoel, J. Am. Chem. Soc., **36**, 2518 (1914) *1*

$CuCO_3$

$C_6H_2(NO_2)_3OH$ **III-976**

When hot concentrated solutions of picric acid and copper carbonate are mixed, copper picrate undecahydrate deposits, on cooling. This is decomposed to the anhydrous salt at 80°. The anhydrous salt forms the tetrahydrate when exposed to moist air at 25°.

$$CuCO_3 + 2C_6H_2(NO_2)_3OH + 10H_2O \rightarrow [C_6H_2(NO_2)_3O]_2Cu \cdot 11H_2O + (CO_2)$$

O. Silberrad and H. A. Phillips, J. Chem. Soc. (London), **93**, 478 (1908) *57*

$CuCO_3$

CrO_3 **III-977**

NH_4OH

Copper carbonate and chromium trioxide solutions were made and mixed and then ammonium hydroxide added to the solution until it is clear. The solution is filtered and allowed to remain in the air. The double chromate salt is formed.

$$(CuCO_3 + 2CrO_3 + 4NH_4OH \rightarrow (NH_4)_2Cu(CrO_4)_2 \cdot 2NH_3 + 3H_2O + CO_2)$$

S. H. C. Briggs, J. Chem. Soc. (London), **83**:1, 394 (1903) *103*

$CuCO_3$

FeS_2 **III-978**

$KHCO_3$

Copper carbonate, marcasite (FeS_2), and potassium bicarbonate, with carbon dioxide in a sealed tube, heated for 14 hours at 160° to 170° react to form cuprous sulfide, ferric oxide, potassium sulfate, carbon dioxide and water.

$$2FeS_2 + 6CuCO_3 + 2KHCO_3 \rightarrow 3Cu_2S + Fe_2O_3 + 8CO_2 + K_2SO_4 + H_2O$$

H. N. Stokes, J. Am. Chem. Soc., **29**, 308 (1907) *13*

$CuCO_3$

$H_2C_4H_4O_5$ **III-979**

$H_3C_6H_5O_7$

Cupric malate is prepared by adding malic acid to cupric carbonate. Cupric citrate is prepared from citric acid in a similar way. The malate is decomposed by the addition of water, forming the acid salt and the basic salt.

(a) $CuCO_3 + H_2C_4H_4O_5 \rightarrow CuC_4H_4O_5 + (H_2O + CO_2)$

(b) $3CuCO_3 + 2H_3C_6H_5O_7 \rightarrow Cu_3(C_6H_5O_7)_2 + (3CO_2 + 3H_2O)$

(c) $3CuC_4H_4O_5 + H_2O \rightarrow C_4H_4O_5 \cdot CuO + CuH_2(C_4H_4O_5)_2$

S. V. Pickering, J. Chem. Soc. (London), **103**, 1355 (1913) *57*

$CuCO_3$

H_2F_2 **III-980**

A basic copper fluoride is formed when copper carbonate is treated with hydrofluoric acid and the resulting salt boiled with water.

(a) $CuCO_3 + H_2F_2 \rightarrow CuF_2 \cdot H_2O + (CO_2)$

(b) $2CuF_2 \cdot H_2O \rightarrow CuO \cdot CuF_2 \cdot H_2O + (H_2F_2)$

Berzelius, Pogg. Ann., **1**, 28 (1824)
Ref., Ann. chim. phys., [2], **24**, 61 (1823)
Ref., Edmister and Copper, J. Am. Chem. Soc., **42**, 2419 (1920) *1*

$CuCO_3$

H_2F_2 **III-981**

K_2CO_3

Copper carbonate is dissolved in hydrofluoric acid. To this solution potassium carbonate is added. Colorless copper potassium fluoride is precipitated.

(a) $2CuCO_3 + 3H_2F_2 \rightarrow 2CuF_2 \cdot HF + 2CO_2 + 2H_2O$

(b) $2CuF_2 \cdot HF + K_2CO_3 \rightarrow \underline{2(CuF_2 \cdot KF)} + H_2O + CO_2$

H. V. Helmoholt, Z. anorg. Chem., **3**, 115 (1893) *28*

$CuCO_3$

H_2F_2 **III-982**

Rb_2CO_3

Copper carbonate is dissolved in hydrofluoric acid. To this solution rubidium carbonate is added. Colorless copper rubidium fluoride is precipitated.

(a) $2CuCO_3 + 3H_2F_2 \rightarrow 2CuF_2 \cdot HF + 2CO_2 + 2H_2O$

(b) $2CuF_2 \cdot HF + Rb_2CO_3 \rightarrow \underline{2(CuF_2 \cdot RbF)} + CO_2 + H_2O$

H. V. Helmoholt, Z, anorg. Chem., **3**, 115 (1893) *28*

$CuCO_3$

H_2SeO_3 **III-983**

Cl_2

Pure copper carbonate is converted into copper selenate when added to a solution of selenious acid which has been treated with chlorine.

(a) $H_2SeO_3 + Cl_2 + H_2O \rightarrow 2HCl + H_2SeO_4$

(b) $2CuCO_3 + 2HCl + H_2SeO_4$
$\rightarrow CuSeO_4 + CuCl_2 + 2CO_2 + 2H_2O$

L. M. Dennis and J. P. Koller, J. Am. Chem. Soc., **41**, 956 (1919) *1*

$CuCO_3$

$H_3C_6H_5O_7$ **III-984**

In the presence of alcohol, addition of citric acid to cupric carbonate causes precipitation of basic cupric citrate.

$$2H_3C_6H_5O_7 + 4CuCO_3 + 2H_2O \rightarrow Cu_3(C_6H_5O_7)_2 \cdot CuO \cdot 5H_2O + 4CO_2$$

S. V. Pickering, J. Chem. Soc. (London), **103**, 1355 (1913) *57*

$CuCO_3 \cdot Cu(OH)_2$

NH_4OH **III-985**

CO_2

When malachite is dissolved in ammonia—ammonium bicarbonate, the reaction is represented by the following equations:

(a) $CuCO_3 \cdot Cu(OH)_2 + CO_2 + 4NH_4OH \rightarrow 2Cu(NH_3)_2 \cdot CO_3 + 5H_2O$

(b) $CuCO_3 \cdot Cu(OH)_2 + 6NH_4OH \rightarrow Cu(NH_3)_2CO_3 + Cu(NH_3)_4(OH)_2 + 6H_2O$

(c) $CuCO_3 \cdot Cu(OH)_2 + 9NH_4OH \rightarrow 2Cu(NH_3)_4(OH)_2 + NH_4HCO_3 + 7H_2O$

E. B. Holland and G. M. Gilligan, J. Phys. Chem., **31**, 731 (1927) *85*

$CuCO_3$

Na_2CO_3 **III-986**

A deep blue solution is formed by dissolving copper carbonate in a solution of sodium carbonate containing sodium bicarbonate. Light blue crystals of a double salt separate on standing.

$$CuCO_3 + Na_2CO_3 + 3H_2O \rightarrow \underline{CuCO_3 \cdot Na_2CO_3 \cdot 3H_2O}$$

Deville, Ann. chem. phys., **33**, 75 (1851)
Ref., S. V. Pickering, J. Chem. Soc. (London), **95**:2, 1411 (1909) *57*

$CuCO_3$

d,l-alanine **III-987**

If copper carbonate is boiled with d,l-alanine in aqueous solution, the copper salt of this acid is formed. It can be precipitated with propyl alcohol almost quantitatively.

$$(CuCO_3 + 2C_3H_7O_2N \rightarrow (C_3H_6O_2N)_2Cu + H_2O + CO_2)$$

P. Pfeiffer and W. Christeleit, Z. physiol. Chem., **245**, 197 (1936) *14*

$CuCO_3$

d,l-lactic acid **III-988**

If copper carbonate is boiled with an aqueous solution of d,l-lactic acid, and if after almost complete evaporation d,l-lactic acid is added again, the copper salt of this acid is formed.

$$(CuCO_3 + 2C_3H_6O_3 \rightarrow C_6H_{10}O_6Cu + H_2O + CO_2)$$

P. Pfeiffer and W. Christeleit, Z. physiol. Chem., **247**, 262, (1937) *14*

$CuCO_3 \cdot Cu(OH)_2$

CO_2 **III-989**

Malachite dissolves in water containing dissolved carbon dioxide to form a bicarbonate.

$$CuCO_3 \cdot Cu(OH)_2 + 3H_2CO_3 \rightarrow 2Cu(HCO_3)_2 + 2H_2O$$

C. A. Seyler, Analyst, **33**, 454 (1908) *40*

$CuCO_3 \cdot Cu(OH)_2$

HCOOH **III-990**

Copper formate is produced when pure basic copper carbonate is treated with 90% formic acid.

$$CuCO_3 \cdot Cu(OH)_2 + 4HCOOH \rightarrow 2(HCOO)_2Cu + (CO_2 + 3H_2O)$$

J. Kendall and H. Adler, J. Am. Chem. Soc., **43**, 1477 (1921) *1*

$Cu(C_2H_3O_2)_2$

$C_4H_4(COONH_4)_2$ **III-991**

A solution of ammonium mucate is treated with a solution of copper acetate as long as a precipitate forms. The precipitate is filtered off and dried. It is copper mucate.

A similar reaction occurs when this reagent is treated with nickel acetate.

$$(C_2H_3O_2)_2Cu + C_4H_4(COONH_4)_2 \rightarrow \underline{C_4H_4(COO)_2Cu} + 2C_2H_3O_2 \cdot NH_4$$

H. Rupe, Ann., **256**, 25 (1890) *20*

$Cu(C_2H_3O_2)_2$

C_5H_5N **III-992**

When pyridine is added to an aqueous or an alcoholic solution of cupric acetate and the solution is evaporated, cupric acetate-pyridine complex separates as emerald-green, six-sided platelets and as short prisms.

$$Cu(C_2H_3O_2)_2 + C_5H_5N \rightarrow Cu(C_2H_3O_2)_2 \cdot C_5H_5N$$

F. Foerster, Ber., **25**, 3420 (1892) *91*

$Cu(C_2H_3O_2)_2$

C_5H_5N **III-993**

Powdered cupric acetate is treated with an excess of pyridine to obtain a blue compound having the formula $Cu(C_2H_3O_2)_2 \cdot 4C_5H_5N$.

$$Cu(C_2H_3O_2)_2 + 4C_5H_5N \rightarrow Cu(C_2H_3O_2)_2 \cdot 4C_5H_5N$$

F. Foerster, Ber., **25**, 3421 (1892) *91*

$Cu(C_2H_3O_2)_2$

C_5H_5N **III-994**

Copper acetate treated with pyridine gives one of two compounds depending upon the temperature. The transition point is 44.7°. The salts were described by Foerster (Ber., **25**, 3416 (1892)).

$$Cu(C_2H_3O_2)_2 + C_5H_5N \rightarrow Cu(C_2H_3O_2)_2 \cdot C_5H_5N$$

J. H. Mathews and E. B. Benger, J. Phys. Chem., **18**, 264 (1914) *7*

$Cu(C_2H_3O_2)_2$

$C_6H_5N(COONH_4)_2$ **III-995**

Ammonium picolin-dicarboxylate is dissolved in water and an aqueous solution of copper acetate added as long as a precipitate forms. Filter off the precipitate and wash with water. Copper picolin-dicarboxylate is formed.

$$C_6H_5N(COONH_4)_2 + Cu(C_2H_3O_2)_2 \rightarrow \underline{C_6H_5N(COO)_2Cu} + 2C_2H_3O_2NH_4$$

S. Altar, Ann., **237**, 193 (1887) *20*

$Cu(C_2H_3O_2)_2$

KI **III-996**

Cuprous iodide is precipitated and iodine liberated when cupric acetate is treated with potassium iodide.

$$2Cu(C_2H_3O_2)_2 + 4KI \rightarrow \underline{2CuI} + 4KC_2H_3O_2 + I_2$$

A. H. Low, J. Am. Chem. Soc., **24**, 1083 (1902) *1*

$Cu(C_2H_3O_2)_2$

NH_3 **III-997**

Cupric acetate reacts with ammonia, in alcoholic solution, to form cupric acetate diammoniate. The diammoniate crystallizes when the solution is evaporated and may be recrystallized from strong alcohol.

$$Cu(C_2H_3O_2)_2 + 2NH_3 \rightarrow Cu(C_2H_3O_2)_2 \cdot 2NH_3$$

F. Foerster, Ber., **25**, 3416 (1892) *91*

$Cu(C_2H_3O_2)_2$

NH_3 **III-998**

NH_4I

When the proper amounts of cupric acetate, ammonia, alcohol and acetic acid are mixed, black crystals of tetrammon-tri cupriammonium iodide are obtained.

(a) $(Cu(C_2H_3O_2)_2 + 2NH_4I \rightarrow Cu(NH_3)_2I_2 + 2HC_2H_3O_2)$

(b) $3Cu(NH_3)_2I_2 + 4NH_3 \rightarrow Cu_3I_6(NH_3)_{10}$

T. W. Richards and G. Oenslager, Am. Chem. J., **17**, 302 (1895) *1*

$Cu(C_2H_3O_2)_2$

NH_4OH **III-999**

NH_4I

Clear, deep-blue crystals of ammoncupri ammonium acetoiodide are produced when cupric acetate is dissolved in ammonia water. To this cooled solution add aqueous acetic acid, ammonium iodide and alcohol.

$$Cu(C_2H_3O_2)_2 + 2NH_4OH + NH_4I \rightarrow Cu(NH_3)_3IC_2H_3O_2(+ 2H_2O + HC_2H_3O_2)$$

T. W. Richards and G. Oenslager, Am. Chem. J., **17**, 298 (1895) *1*

$Cu(C_2H_3O_2)_2$

N_2H_4 **III-1000**

Light

An ammoniacal solution of 1% copper acetate to which has been added about 15 to 20 mg of hydrazine hydrate per 10 ml. of solution is reduced by long exposure to light. The reddish product seems to be a mixture of copper and cuprous oxide.

$$Cu(C_2H_3O_2)_2 + N_2H_4 \rightarrow Cu_2O + Cu + \text{(other products)}$$

C. W. Bennett, J. Phys. Chem., **16**, 782 (1912) *7*

$Cu(C_2H_3O_2)_2$

Na_2Te **III-1001**

Normal copper telluride forms as a black precipitate when sodium telluride reacts with copper acetate in presence of acetic acid.

$$Cu(C_2H_3O_2)_2 + Na_2Te \rightarrow \underline{CuTe} + (2NaC_2H_3O_2)$$

C. A. Tibbals, Jr., J. Am. Chem. Soc., **31**, 909 (1909) *1*

$Cu(C_2H_3O_2)_2$

Na_4Te_3 **III-1002**

Anhydrous copper telluride is formed when sodium polytelluride reacts with copper acetate.

$$2Cu(C_2H_3O_2)_2 + Na_4Te_3 \rightarrow \underline{Cu_2Te_3} + (4NaC_2H_3O_2)$$

C. A. Tibbals, Jr., J. Am. Chem. Soc., **31**, 910 (1909) *1*

$Cu(C_2H_3O_2)_2$

SO_2 **III-1003**

A colorless crystalline salt, hydrated copper sulfite, is obtained when sulfur dioxide is passed into a solution of copper acetate. This hemihydrate is converted into the red monohydrate by aqueous sulfurous acid.

(a) $4Cu(C_2H_3O_2)_2 + 11SO_2 + H_2O \rightarrow 2[Cu_2SO_3 \cdot \frac{1}{2}H_2O] + (16CO_2 + 12H_2 + 9S)$

(b) $2[Cu_2SO_3 \cdot \frac{1}{2}H_2O] + H_2O \rightarrow 2Cu_2SO_3 \cdot H_2O$

Ramberg, Z. Physik. Chem., **69**, 512
Ref., J. L. Howe, J. Am. Chem. Soc., **33**, 168 (1911) *1*

$Cu(C_2H_3O_2)_2$

Δ **III-1004**

A water solution of cupric acetate when heated to 170° changes to cuprous oxide, free acetic acid, and CO_2.

$2Cu(C_2H_3O_2)_2 + 5H_2O \rightarrow 2C_2H_4O_2 + Cu_2O + 4CO_2 + (7H_2)$

Riban,
Ref., A. Henninger, Ber., **9**, [2], 1133 (1876) *26*

$Cu(C_2H_3O_2)_2$

3-Amino-4-bromobenzoic acid **III-1005**

3-Amino-4-bromobenzoic acid is dissolved in ammonia and the solution evaporated to dryness to remove excess ammonia, dissolved in water and an aqueous solution of cupric acetate added. The precipitated cupric-3-amino-4-bromobenzoate is washed with water and dried.

A similar reaction occurs when this reagent is treated in a similar manner with lead acetate.

$2C_6H_3NH_2BrCOOH + Cu(C_2H_3O_2)_2 \rightarrow \underline{Cu(C_6H_3NH_2BrCOO)_2} + 2CH_3COOH$

H. Hübner, Ann., **222**, 180 (1884) *20*

$Cu(C_2H_3O_2)_2$

Ammonium-3,4-dibromobenzoate **III-1006**

Ammonium-3,4-dibromobenzoate is dissolved in water and an aqueous solution of cupric acetate added as long as a precipitate forms. The precipitate is filtered off and dried. It is cupric-3,4-dibromobenzoate.

A similar reaction occurs when this reagent is treated with a solution of silver nitrate.

$$2C_6H_3Br_2COONH_4 + Cu(C_2H_3O_2)_2 \rightarrow \underline{Cu(C_6H_3Br_2COO)_2} + 2NH_4C_2H_3O_2$$

H. Hübner, Ann., **222**, 186 (1884) *20*

$Cu(C_2H_3O_2)_2$

p-Nitrophenylalanine **III-1007**

One gram of p-nitrophenylalanine is dissolved in 80 cc. of hot water and a hot aqueous solution of 0.5 gram of cupric acetate in 20 cc. water added. Copper p-nitrophenylalanate with $4H_2O$ precipitates.

$$2C_8H_9N_2O_2COOH + Cu(C_2H_3O_2)_2 \rightarrow \underline{Cu(C_8H_9N_2O_2COO)_2} + 2HC_2H_3O_2$$

E. Erlenmeyer and A. Lipp, Ann., **219**, 223 (1883) *20*

$Cu(C_2H_3O_2)_2$

Phenylalanine **III-1008**

Dissolve 1 g. of phenylalanine in 70 cc. of boiling water and add a hot solution of 0.7 g. of cupric acetate in 20 cc. water. Filter while hot and cool. Cupric phenylalanate with $2H_2O$ precipitates.

$$2C_8H_{10}NCOOH + Cu(C_2H_3O_2)_2 \rightarrow \underline{Cu(C_8H_{10}NCOO)_2} + 2CH_3COOH$$

E. Erlenmeyer and A. Lipp, Ann., **219**, 200 (1883) *20*

$Cu(C_2H_3O_2)_2$

Sodium metaaminobenzoate **III-1009**

An aqueous solution of sodium metaaminobenzoate is treated with an aqueous solution of cupric acetate as long as a precipitate forms, the solution filtered and the residue dried. It is cupric metaaminobenzoate.

$$2C_6H_4NH_2COONa + (C_2H_3O_2)_2Cu \rightarrow (C_6H_4NH_2COO)_2Cu + 2C_2H_3O_2Na$$

H. Hübner, Ann., **195**, 30 (1879) *20*

$Cu(C_2H_3O_2)_2 \cdot C_5H_5N$

Δ **III-1010**

The transition point between the two salts given below is 44.7°.

$$Cu(C_2H_3O_2)_2 \cdot 4C_5H_5N \rightleftarrows \underline{Cu(C_2H_3O_2)_2 \cdot C_5H_5N} + 3C_5H_5N$$

Foerster, Ber., **25**, 3416 (1892)
Ref., J. H. Mathews and E. B. Benger, J. Phys. Chem., **18**, 264 (1914) *7*

$Cu(C_2H_3O_2)_2 \cdot H_2O$

Benzoyl carbinol **III-1011**

If solid copper acetate is shaken for several days with benzoyl carbinol in alcohol and water, cuprous oxide precipitates with simultaneous formation of phenylglyoxal.

$$2Cu(C_2H_3O_2)_2 \cdot H_2O + C_6H_5COCH_2OH \rightarrow \underline{Cu_2O} + 4CH_3COOH + C_6H_5COCHO + H_2O$$

M. Henze, Z. physiol. Chem., **198**, 82 (1931) *14*

$Cu(C_2H_3O_2)_2 \cdot 2NH_3$

NH_3 **III-1012**

When gaseous ammonia is passed over solid cupric acetate diammoniate, heat is liberated, and the solid increases in weight by about 12.80%. The unstable tetrammoniate is probably formed.

$$Cu(C_2H_3O_2)_2 \cdot 2NH_3 + 2NH_3 \rightarrow Cu(C_2H_3O_2)_2 \cdot 4NH_3$$

F. Foerster, Ber., **25**, 3419 (1892) *91*

$CuC_6H_4SO_4$

C_5H_5N **III-1013**

Olive green colored needles are obtained when pyridine is added to a conc. solution of copper o-phenol sulfonate.

$$C_6H_4SO_4Cu + 2C_5H_5N + H_2O \rightarrow C_6H_4SO_4Cu \cdot (C_5H_5N)_2 \cdot H_2O$$

H. Ley and O. Erler, Z. anorg. Chem., **56**, 401 (1908) *28*

$CuC_6H_4SO_4$

NH_4OH **III-1014**

To a saturated solution of o-phenol copper sulfonate, an excess of ammonium hydroxide is added. A deep green solution will result. After some time, prismatic needles will be formed.

$$C_6H_4SO_4Cu + 2NH_4OH + H_2O \rightarrow C_6H_6SO_5 \cdot Cu(NH_3)_2 \cdot 2H_2O$$

H. Ley and O. Erler, Z. anorg. Chem., **56**, 401 (1908) *28*

$CuC_6H_4SO_4$

NH_4OH **III-1015**

To a concentrated solution of copper p-phenol sulfonate, an excess of conc. ammonium hydroxide is added. Deep dark blue needles will be formed.

$$2C_6H_4SO_4Cu + 4NH_4OH \rightarrow C_{12}H_{10}S_2O_8 \cdot Cu(NH_3)_4 \cdot 2(H_2O) + ?$$

H. Ley and O. Erler, Z. anorg. Chem., **56**, 401 (1908) *28*

$Cu(C_6H_5 \cdot COO)_2$

NH_3 **III-1016**

Copper benzoate will react at room temperature with ammonia gas. An excess of ammonia at low temperature will yield a dark blue colored precipitate. Finally one more molecule of ammonia may be added yielding copper benzoate pentammoniate of green color.

(a) $Cu(C_6H_5 \cdot COO)_2 + 3NH_3 \rightarrow \underline{Cu(C_6H_5 \cdot COO)_2 \cdot 3NH_3}$

(b) $Cu(C_6H_5 \cdot COO)_2 \cdot 3NH_3 + NH_3 \rightarrow \underline{Cu(C_6H_5 \cdot COO)_2 \cdot 4NH_3}$

(c) $Cu(C_6H_5 \cdot COO)_2 \cdot 4NH_3 + NH_3 \rightarrow \underline{Cu(C_6H_5 \cdot COO)_2 \cdot 5NH_3}$

Fritz Ephraim and Edward Bolle, Ber., **48**, 1770 (1916) *28*

$Cu(C_6H_5SO_4)_2$

C_5H_5N

III-1017

To a saturated solution of copper p-phenol sulfonate, pyridine is added. A blue colored salt will be obtained.

$$(C_6H_5SO_4)_2Cu + 4C_5H_5N \rightarrow C_{12}H_{16}S_2O_8Cu \cdot 4(C_5H_5N)$$

H. Ley and O. Erler, Z. anorg. Chem., **56**, 401 (1908) *28*

$Cu(C_6H_{11}O_2)_2$

Pb

III-1018

A solution of copper capronate in pyridine (which has a green color) turns immediately brown on contact with metallic lead and a deposit of metallic copper is formed simultaneously on the surface of the lead.

$$Cu(C_6H_{11}O_2)_2 + Pb \rightarrow Pb(C_6H_{11}O_2)_2 + \underline{Cu}$$

J. L. Sammis, J. Phys. Chem., **10**, 593 (1906) *7*

$Cu(C_7H_5O_3)_2$

C_5H_5N

III-1019

Blue needles are formed when pyridine is added to an alcoholic copper salicylate solution.

$$(C_7H_5O_3)_2Cu + 2C_5H_5N \rightarrow C_{24}H_{20}N_2O_6Cu$$

H. Ley and O. Erler, Z. anorg. Chem., **56**, 401 (1908) *28*

$Cu(C_7H_5O_3)_2$

NH_3

III-1020

A complex salt of dark blue color is formed when conc. ammonia is added to copper p-oxybenzoate.

$$(C_7H_5O_3)_2Cu + 2NH_3 + 5H_2O \rightarrow C_{14}H_{16}O_6N_2Cu \cdot 5H_2O$$

H. Ley and O. Erler, Z. anorg. Chem., **56**, 401 (1908) *28*

$CuC_7H_6SO_4$

C_5H_5N

III-1021

Dark olive green colored needles are formed when pyridine is added to a solution of copper o-cresol-o-sulfonate solution.

$$CuC_7H_6SO_4 + 2C_5H_5N + H_2O \rightarrow C_{17}H_{18}O_5N_2SCu$$

H. Ley and O. Erler, Z. anorg. Chem., **56**, 401 (1908) *28*

$CuC_7H_6O_4S$

C_5H_5N **III-1022**

To a solution of copper o-cresol-p-sulfonate in alcohol and water, pyridine is added in excess. Dark blue colored needles are formed.

$$2C_7H_6SO_4Cu + 4C_5H_5N + H_2O \rightarrow C_{34}H_{34}O_8N_4S_2Cu + (CuO)$$

H. Ley and O. Erler, Z. anorg. Chem., **56**, 401 (1908) *28*

$CuC_7H_6SO_4$

NH_3 **III-1023**

A dark green colored salt is obtained when an excess of ammonia is added to a solution of copper o-cresol-o-sulfonate.

$$C_7H_6SO_4Cu + 2NH_3 + H_2O \rightarrow C_7H_{14}O_5N_2SCu$$

H. Ley and O. Erler, Z. anorg. Chem., **56**, 401 (1908) *28*

$CuC_7H_6SO_4$

NH_3 **III-1024**

Blue needles are formed when ammonia solution is added to copper o-cresol-p-sulfonate.

$$Cu2C_7H_6SO_4 + 4NH_3 + 3H_2O \rightarrow C_{14}H_{26}O_8S_2N_4Cu \cdot 2(H_2O) + CuO$$

H. Ley and O. Erler, Z. anorg. Chem., **56**, 401 (1908) *28*

$Cu(C_{16}H_{31}O_2)_2$

Pb **III-1025**

A toluene solution of copper palmitate, at the boiling point reacts with metallic lead, producing in two hour's time a thick black scale on the lead. In similar procedures, some tarnish is observed on bismuth but there is no effect on magnesium, antimony, cobalt, silver, zinc, tin or cadmium. In pyridine solutions, the reaction with lead is rapid. This replacement proceeds also in the absence of solvent at temperatures in the range, 125–140°.

$$Cu(C_{16}H_{31}O_2)_2 + Pb \rightarrow \underline{Cu} + Pb(C_{16}H_{31}O_2)_2$$

J. L. Sammis, J. Phys. Chem., **10**, 593 (1906) *7*

$Cu(C_{18}H_{33}O_2)_2$

Cd

Pb

III-1026

Cupric oleate in amyl nitrate reacts at boiling temperatures with lead and cadmium, coating these metals with a bright red metallic deposit. Bismuth is somewhat tarnished but silver, magnesium, cobalt, aluminum, tin and iron are unaffected. The reactions were observed over a period of two hours. These reactions have been studied using benzene, carbon disulfide ethyl acetate, acetone, carbon tetrachloride, benzylamine, pyridine, etc. as solvents and also zinc, nickel, bismuth, antimony, and platinum as metals.

(a) $Cu(C_{18}H_{33}O_2)_2 + Cd \rightarrow \underline{Cu} + Cd(C_{18}H_{33}O_2)_2$

(b) $Cu(C_{18}H_{33}O_2)_2 + Pb \rightarrow \underline{Cu} + Pb(C_{18}H_{33}O_2)_2$

J. L. Sammis, J. Phys. Chem., **10**, 593 (1906)

7

$Cu(C_{18}H_{33}O_2)_2$

$SnCl_4$

III-1027

A slight excess of stannic chloride in dry benzene will precipitate copper chloride immediately and completely from a solution of copper oleate in benzene. The precipitate is brown and somewhat contaminated by stannic oleate. The stannic oleate left in solution may be recovered by evaporating the solvent and excess stannic chloride. Phosphorus trichloride, arsenic trichloride and silicon tetrachloride may be substituted for the tin chloride with similar rapid and quantitative precipitation of copper chloride.

(a) $2Cu(C_{18}H_{33}O_2)_2 + SnCl_4 \rightarrow Sn(C_{18}H_{33}O_2)_2 + \underline{2CuCl_2}$

(b) $3Cu(C_{18}H_{33}O_2)_2 + 2PCl_3 \rightarrow 2P(C_{18}H_{33}O_2)_3 + \underline{3CuCl_2}$

(c) $3Cu(C_{18}H_{33}O_2)_2 + 2AsCl_3 \rightarrow 2As(C_{18}H_{33}O_2)_3 + \underline{3CuCl_2}$

(d) $2Cu(C_{18}H_{33}O_2)_2 + SiCl_4 \rightarrow Si(C_{18}H_{33}O_2)_2 + \underline{2CuCl_2}$

L. Kahlenberg, J. Phys. Chem., **6**, 1 (1902)

7

CuC_2O_4

C_5H_5N **III-1028**

When copper oxalate is mixed with pyridine, sky-blue, prismatic crystals are formed. The composition of the crystals indicates the formula given below.

$$CuC_2O_4 + 2C_5H_5N \rightarrow CuC_2O_4 \cdot 2C_5H_5N$$

K. Seubert and G. Rauter, Ber., **25**, 2825 (1892) *91*

CuC_2O_4

NH_3 **III-1029**

When copper oxalate is treated with an excess of aqueous ammonia, a blue solution is obtained. When the solution evaporates, dark-blue triclinic crystals form. The composition of the crystals corresponds to the formula shown below.

$$(CuC_2O_4 + 2NH_3 + 2H_2O \rightarrow CuC_2O_4 \cdot (NH_3)_2 \cdot 2H_2O)$$

K. Seubert and G. Rauter, Ber., **25**, 2823 (1892) *91*

CuCl

C_2H_2 **III-1030**

The reaction of acetylene with cuprous chloride may be used for purifying the acetylene.

$$(2CuCl + C_2H_2 \leftrightarrows \underline{Cu_2C_2} + 2HCl)$$

Römer, Ann., **233**, 183 (1886)
Ref., K. Stahrfoss, J. Chim. phys., **16**, 175 (1918) *69*

CuCl

C_2H_2 **III-1031**

By passing acetylene into an ammoniacal solution of cuprous chloride, a flocculent, reddish brown precipitate of cuprous acetyl oxide is formed.

$$4CuCl + 2C_2H_2 + 2H_2O \rightarrow \underline{2HC_2Cu_2O} + (4HCl + H_2)$$

Berthelot, Compt. rend., **62**, 456 (1866) *25*

CuCl

C_2H_2

KCl **III-1032**

By passing acetylene into a concentrated solution of cuprous chloride in potassium chloride, a yellow precipitate is formed consisting of a double chloride. The latter upon washing with a saturated solution of potassium chloride, gives cupro acetyl chloride.

(a) $2CuCl + C_2H_2 + KCl \rightarrow (\underline{HC_2Cu_2Cl \cdot KCl} + HCl)$

(b) $HC_2Cu_2Cl \cdot KCl \rightarrow \overline{HC_2Cu_2Cl + KCl}$

Bertholet, Compt. rend., **62**, 456 (1866) *29*

CuCl

$C_2H_5 \cdot S \cdot CH_2COOH$ **III-1033**

Cuprous chloride dissolves in ethylthioglycolic acid and yields on evaporation colorless, flat prisms of anhydrous ethylthioglycolic acid cuprous chloride. If a 1:1 acid-water mixture is used, the mono-hydrated compound is produced.

(a) $CuCl + 2C_2H_5 \cdot S \cdot CH_2COOH \rightarrow (C_2H_5 \cdot S \cdot CH_2COOH)_2 \cdot CuCl$

(b) $2CuCl + C_2H_5 \cdot S \cdot CH_2COOH + H_2O$
$\rightarrow C_2H_5 \cdot S \cdot CH_2COOH \cdot (CuCl)_2 \cdot H_2O$

L. Ramberg, Act. Univ. Lund. Med. Matem. Natur., N. S. **4**, No. 2, 4 (1908) *10*

CuCl

$C_2H_5 \cdot S \cdot CH_2COOH$ **III-1034**

KSCN

Cuprous chloride is dissolved in ethylthioglycolic acid, potassium thiocyanate is dissolved in ethylthioglycolic acid and the two warm solutions are mixed. Some anhydrous ethylthioglycolic acid cuprous thiocyanate separates as small, almost microscopic, colorless, oblique tabular crystals.

$CuCl + KSCN + C_2H_5 \cdot S \cdot CH_2COOH$
$\rightarrow CuSCN \cdot C_2H_5 \cdot S \cdot CH_2COOH + (KCl)$

L. Ramberg, Acta Univ. Lund. Med. Matem. Natur., N. S. **4**, No. 2, 15 (1908) *10*

CuCl

C_5H_5N **III-1035**

Upon addition of copper chloride to pyridine the complex precipitates as a green solid.

$$2CuCl + 4C_5H_5N \rightarrow \underline{(Cu_2(C_5H_5N)_4)Cl_2}$$

D. O. Marouhian, J. Gen. Chem. (USSR), **10**, 917 (1940) *60*

CuCl

$C_{18}H_{12}N_2$ **III-1036**

2,2′-Biquinolyl, prepared by catalytic dehydrogenation of quinoline, gives a purple colored compound with the cuprous ion. The reaction is very sensitive and is specific for copper.

$$(CuCl + 2C_{18}H_{12}N_2 \rightarrow CuCl \cdot (C_{18}H_{12}N_2)_2)$$

J. G. Breckenridge, R. W. J. Lewis and L. A. Quinck, Can. J. Res., **B17**, 258 (1939) *69*

CuCl

CuNi **III-1037**

ε

On passing current through a cell having a copper-nickel anode and cuprous chloride solution, copper is deposited on the cathode and nickel is dissolved.

$$2CuCl + CuNi \rightarrow 3Cu + NiCl_2$$

J. W. Richards, Trans. Am. Eectrochem. Soc., **5**, 91 (1904) *8*

CuCl

HCl **III-1038**

O_2

If cuprous chloride, heated between 100° and 200°, is treated with hydrochloric acid, drop by drop, in the presence of air, it is transformed into cupric chloride and water vapor is given off. The same reaction takes place when gaseous HCl is used.

$$4CuCl + 4HCl + O_2 \rightarrow 4CuCl_2 + 2H_2O$$

A. Mallet, Compt. rend., **66**, 350 (1868) *29*

CuCl

H_2O

III-1039

Freshly precipitated cuprous chloride washed with water forms hydrochloric acid in solution and a precipitate of cuprous oxide.

$$2CuCl + H_2O \rightarrow 2HCl + \underline{Cu_2O}$$

J. R. Haywood, J. Phys. Chem., **1**, 411 (1896–7) *7*

CuCl

H_2O

III-1040

Cuprous chloride will be decomposed by water either to cuprous hydroxide and hydrochloric acid or to cupric chloride and metallic copper.

(a) $$CuCl + H_2O \rightarrow \underline{CuOH} + HCl$$

(b) $$2CuCl \rightarrow CuCl_2 + \underline{Cu}$$

G. Bodländer and O. Starbeck, Z. anorg. Chem., **31**, 1 (1902) *28*

CuCl

H_2O

O_2

III-1041

When cuprous chloride is heated at 500°C in a stream of moisture and air, cupric oxide and hydrogen chloride are formed.

$$4CuCl + 2H_2O + O_2 \rightarrow 4CuO + 4HCl$$

P. Klason, Sv. Kem. Tidsk., **29**, 210 (1917) *10*

CuCl

H_2O

SO_2

ϵ

III-1042

If an electric current is passed through a solution of cuprous chloride treated with sulfur dioxide in an electrolyzer the following reactions take place.

(a) $$2CuCl + \epsilon \rightarrow 2Cu + Cl_2$$

(b) $$2Cl + SO_2 + 2H_2O \rightarrow 2HCl + H_2SO_4$$

Greenwalt, J. Ind. Eng. Chem., **2**, 555 (1910) *22*

CuCl

H_2S **III-1043**

Hydrogen chloride is liberated when cuprous chloride reacts with liquid hydrogen sulfide.

$$2CuCl + H_2S \rightarrow 2HCl + Cu_2S$$

A. W. Ralston and J. A. Wilkinson, J. Am. Chem. Soc., **50**, 259 (1928) *1*

CuCl

KCl **III-1044**

Cuprous chloride will react with diluted solutions of potassium chloride yielding complex potassium copper chloride salts.

(a) $$CuCl + KCl \rightarrow KCuCl_2$$

(b) $$CuCl + 2KCl \rightarrow K_2CuCl_3$$

G. Bodländer and O. Starbeck, Z. anorg. Chem., **31**, 1 (1902) *28*

CuCl

$K_3Cr(CN)_6$ **III-1045**

Cuprous chromicyanide is formed as an orange-yellow precipitate when a solution of cuprous chloride is added to a solution of potassium chromicyanide.

$$3CuCl + K_3Cr(CN)_6 \rightarrow \underline{Cu_3(Cr(CN)_6)} + (3KCl)$$

F. V. D. Cruser and E. H. Miller, J. Am. Chem. Soc., **28**, 1139 (1906) *1*

CuCl

MgO **III-1046**

When solid cuprous chloride and magnesium oxide stand in contact with water, magnesium chloride and cuprous hydroxide are formed.

$$2CuCl + MgO + H_2O \rightarrow MgCl_2 + 2CuOH$$

Sterry Hunt,
Ref., Ch. Friedel, Ber., **3**, 35 (1870) *11*

CuCl("Cu_2Cl")

NH_3 **III-1047**

In the dry method, ammonia gas forms three additional compounds with cuprous chloride: (a) is formed on slight warming; (b) is formed in the cold; (c) is formed on prolonged action, but is difficult to obtain in the pure state and the formula is uncertain. In the wet method, cuprous chloride is boiled in a solution of hydrochloric acid to which a slight excess of ammonia has been added, reacting as in (d).

(a) $$2CuCl + 2NH_3 \rightarrow 2CuCl \cdot NH_3$$

(b) $$CuCl + NH_3 \rightarrow CuCl \cdot NH_3$$

(c) $$CuCl + 2NH_3 \rightarrow CuCl \cdot 2NH_3$$

(d) $$CuCl + NH_3 + 2H_2O \rightarrow CuCl \cdot NH_3 \cdot 2H_2O$$

P. P. Deherain, Compt. rend., **55**, 807 (1862) *29*

CuCl

NaOH **III-1048**

Hydrated cuprous oxide is precipitated when a hydrochloric acid solution of cuprous chloride is added to a dilute sodium hydroxide solution.

$$8CuCl + 8NaOH \rightarrow \underline{4(Cu_2O) \cdot H_2O} + 8NaCl$$

H. V. Tartar and W. L. Semon, J. Am. Chem. Soc., **43**, 494 (1921) *1*

CuCl

$Na_2S_2O_3$ **III-1049**

Cuprous chloride and sodium thiosulfate when mixed produce dicuprous thiosulfate disodium thiosulfate dicuprous chloride.

$$6CuCl + 4Na_2S_2O_3 \rightarrow 2[(Cu_2S_2O_3) \cdot (Na_2S_2O_3) \cdot (CuCl)] + (4NaCl)$$

E. H. Riesenfeld and E. Grünthal, Med. K. Vet. Nobelinst., **6**, No. 9, 32 (1924) *10*

CuCl

$Na_4Fe(CN)_6$ **III-1050**

$K_4Fe(CN)_6$

By boiling cuprous chloride with a solution of sodium ferrocyanide, colorless crystals of sodium cuproferrocyanide are obtained. The same type reaction occurs with potassium ferrocyanide.

(a) $2CuCl + Na_4Fe(CN)_6 \rightarrow \underline{Na_2Cu_2Fe(CN)_6} + 2NaCl$

(b) $2CuCl + K_4Fe(CN)_6 \rightarrow \overline{K_2Cu_2Fe(CN)_6} + 2KCl$

J. Messner, Z. anorg. Chem., **8**, 368 (1895) *28*

CuCl

O_2 **III-1051**

Cuprous chloride is readily oxidized by atmospheric oxygen to form copper oxide and copper chloride.

$$4CuCl + O_2 \rightarrow 2CuO + 2CuCl_2$$

L. P. Curtin, Ind. Eng. Chem., **19**, 993 (1927) *23*

CuCl

O_2 **III-1052**

When cuprous chloride is heated at 500°C in a stream of dry air, cupric oxide and chlorine are formed.

$$2CuCl + O_2 \rightarrow 2CuO + Cl_2$$

P. Klason, Sv. Kem. Tidsk., **29**, 210 (1917) *10*

CuCl

O_2 **III-1053**

Cuprous chloride absorbs oxygen from the air spontaneously. At ordinary temperature, in moist air, the reaction is complete in several hours. At higher temperatures (100–200°), in the presence of water vapor, the reaction is almost instantaneous.

$$(4CuCl + O_2 + 5H_2O \rightarrow CuCl_2 \cdot 3Cu(OH)_2 \cdot H_2O + 2HCl)$$

A. Mallet, Compt. rend., **66**, 349 (1868)
Ref., Proust, J. de Physique, **51**, 181 (1800)
Ref., M. Groger, Z. anorg. Chem., **28**, 154 (1901) *29*

CuCl

O_2 **III-1054**

Cu_2O

Cuprous chloride reacts with oxygen to form mixtures of cupric oxide, cupric chloride and cupric oxychloride depending on the amount of oxygen present.

(a) $$4CuCl + O_2 \rightarrow 2CuCl_2 + 2CuO$$

(b) $$3Cu_2O + 2CuCl_2 + 8H_2O + 3O \rightarrow 2Cu_4(OH)_6Cl_2 \cdot H_2O$$

G. D. Bengough and R. May, 7th report of the institute of metals to the committee on corrosion.
Ref., Can. Chem. & Met., **8**, 267 (1924) *15*

CuCl

P_2S_5 **III-1055**

Cuprous thiophosphate is obtained when a mixture of cuprous chloride and phosphorus pentasulfide is heated.

$$3CuCl + P_2S_5 \rightarrow \underline{Cu_3PS_4} + PSCl_3$$

E. Glatzel, Z. anorg. Chem., **4**, 186 (1893) *28*

$CuCl \cdot NH_3$

HCl **III-1056**

The cuprous chloride ammonia compounds add on a mole of hydrochloric acid.

(a) $$2CuCl \cdot NH_3 + 2HCl \rightarrow 2CuCl \cdot NH_4Cl$$

(b) $$CuCl \cdot NH_3 + HCl \rightarrow CuCl \cdot NH_4Cl$$

P. P. Deherain, Compt. rend., **55**, 807 (1862) *29*

$Cu(ClO_3)_2$

NH_3 **III-1057**

An aqueous solution of copper chlorate (30–40%) is treated in a small flask with gaseous ammonia. The color changes to a deep-blue. On adding alcohol needles are formed which melt on heating, and eventually detonate violently.

$$Cu(ClO_3)_2 + 4NH_3 \rightarrow \underline{Cu(ClO_3)_2 \cdot 4NH_3}$$

F. Ephraim and A. Jansen, Ber., **48**, 46 (1915) *28*

$Cu(ClO_3)_2 \cdot (NH_3)_4$

NH_3 **III-1058**

Ammonia gas is passed over dry copper chlorate tetrammoniate at room temperature. Copper chlorate hexammoniate of ultramarine-blue color is formed.

$$Cu(ClO_3)_2 \cdot 4NH_3 + 2NH_3 \rightarrow \underline{Cu(ClO_3)_2 \cdot 6NH_3}$$

F. Ephraim and A. Jansen, Ber., **48**, 48 (1915) *28*

$CuCl_2$

Ag **III-1059**

Cupric chloride when warm and in the presence of other metal chlorides, reacts readily with silver and its compounds to form silver chloride.

$$CuCl_2 + Ag \rightarrow CuCl + \underline{AgCl}$$

Greenwalt, J. Ind. Eng. Chem., **2**, 555 (1910) *22*

$CuCl_2$

Ag_2S **III-1060**

Silver sulfide reacts with a cupric chloride solution, precipitating sulfur and silver chloride and leaving cuprous chloride in solution.

$$Ag_2S + 2CuCl_2 \rightarrow \underline{S} + \underline{2AgCl} + 2CuCl$$

A. Larsson, Sv. Kem. Tidsk., **4**, 86 (1892) *10*

$CuCl_2$

CH_3COONa **III-1061**

When a hot solution of cupric chloride reacts with sodium acetate, a blue-greenish precipitate of basic copper chloride is obtained.

$$4CuCl_2 + 6CH_3COONa + 3H_2O$$
$$\rightarrow \underline{CuCl_2 \cdot 3CuO} + 6NaCl + 6CH_3COOH$$

W. Casselmann, Z. anal. Chem., **4**, 24 (1865) *28*

$CuCl_2$

$CH_3 \cdot C(:S) \cdot NH_2$ **III-1062**

If an alcoholic solution of thio-acetamide be added to a solution of cupric chloride, a white precipitate of the corresponding chloromercaptide is formed; hydrochloric acid is also formed.

$$CuCl_2 + CH_3 \cdot C(:S) \cdot NH_2 \rightarrow \underline{CH_3 \cdot C(:NH) \cdot S \cdot CuCl} + HCl$$

Prafulla C. Rây, J. Chem. Soc. (London), **115**:2, 874 (1919) *48*

$CuCl_2$

$(CH_3)_2S$ **III-1063**

Cuprous chloride methyl sulfide forms when methyl sulfide is added to a concentrated solution of cupric chloride.

$$2(CH_3)_2S + 2CuCl_2 \rightarrow \underline{2CuCl(CH_3)_2S} + (Cl_2)$$

F. C. Phillips, J. Am. Chem. Soc., **23**, 255 (1901) *1*

$CuCl_2$

$C:S(NH_2)_2$ **III-1064**

If an alcoholic solution of thio-carbamide be added with constant stirring to an alcoholic solution of cupric chloride, a granular precipitate (which is white after drying in vacuuo) of the compound shown below is formed.

$$C:S(NH_2)_2 + CuCl_2 \rightarrow \underline{NH_2 \cdot C(:NH) \cdot S \cdot CuCl} + HCl$$

Prafulla C. Rây, J. Chem. Soc. (London), **115**:2, 873 (1919) *48*

$CuCl_2$

C_2H_2 **III-1065**

Colorless crystals are obtained by the reaction of an absolute alcoholic solution of copper chloride with acetylene.

$$6CuCl_2 + C_2H_2 \rightarrow (Cu_2Cl_2)_3 \cdot C_2H_2 + ?$$

K. A. Hofmann and F. Kuespert, Z. anorg. Chem., **15**, 204 (1897) *28*

$CuCl_2$

C_2H_5OH **III-1066**

An alcoholic solution of cupric chloride becomes cuprous chloride when exposed to light.

$$2CuCl_2 + C_2H_5OH \rightarrow 2CuCl + (CH_3CHO + 2HCl)$$

F. P. Venable, J. Am. Chem. Soc., **21**, 223 (1899) *1*

$CuCl_2$

$(C_2H_5)_3SI$ **III-1067**

Triethyl sulfonium cupric iodide precipitates as a red brown oil when a saturated solution of cupric chloride is added to a saturated solution of triethyl sulfonium iodide.

$$CuCl_2 + 4(C_2H_5)_3SI \rightarrow \underline{(S(C_2H_5)_3I)_2CuI_2} + (2(C_2H_5)_3SCl)$$

R. L. Datta and T. Ghosh, J. Am. Chem. Soc., **36**, 1020 (1914) *1*

$CuCl_2$

$C_3H_3NS_2O$ **III-1068**

$C_{11}H_{12}ON_4S$

$C_{11}H_{13}ON_3S$

Copper chloride reacts with rhodamine, yielding a green precipitate. The same type reaction occurs with 5-para-dimethylaminophenylimino-2-thiohydantoine and 5-para-dimethylaminobenzal-2-thiohydantoine.

(a) $CuCl_2 + 2C_3H_3NS_2O + H_2O$
$\rightarrow \underline{(C_3H_2NS_2O)CuCl \cdot C_3H_4NS_2O_2} + (2HCl)$

(b) $CuCl_2 + C_{11}H_{12}ON_4S + H_2O \rightarrow \underline{C_{11}H_{10}ON_4SCuH_2O} + (2HCl)$

(c) $2CuCl_2 + 2C_{11}H_{13}ON_3S + 11H_2O$
$\rightarrow \underline{2C_{11}H_{11}ON_3SCu \cdot 11H_2O} + 4HCl$

I. V. Dubsky, V. Lindelar and V. Cernak, Mikrochem., **25**, 124 (1938) *28*

$CuCl_2$

$C_3H_{10}ON_2$ **III-1069**

Cupric salts form coordination compounds with 1:3 diamino isopropanol in ethanol solution. Zinc chloride and silver nitrate form similar complexes:

$$(CuCl_2 + 2C_3H_{10}ON_2 \rightarrow CuCl_2 \cdot (C_3H_{10}ON_2)_2)$$

F. G. Mann, J. Chem. Soc. (London), 2904 (1927)
Ref., J. G. Breckenridge and J. W. R. Hodgins, Can. J. Res., **B17**, 331 (1939) *69*

$CuCl_2$

C_5H_5N **III-1070**

From a study of equilibrium curves for cupric chloride-pyridine systems, the transition point between the two compounds $CuCl_2 \cdot 2C_5H_5N$ and $CuCl_2 \cdot 3C_5H_5N$ is found to be about 58°. Below −10°, a new solid phase appears, the composition of which corresponds to the composition, $CuCl_2 \cdot 6C_5H_5N$.

$$CuCl_2 + 6C_5H_5N \rightarrow CuCl_2 \cdot 6C_5H_5N$$

J. A. Mathews and S. Spero, J. Phys. Chem., **21**, 402 (1917) *7*

$CuCl_2$

$C_8H_6N_2S_2$ **III-1071**

NH_3

Copper chloride will react with benzenylazosulfim in presence of ammonia yielding an insoluble copper salt.

$$CuCl_2 + 2C_8H_6N_2S_2 + 2NH_3 \rightarrow \underline{Cu(C_8H_5N_2S_2)_2} + 2NH_4Cl$$

J. V. Dubsky, A. Okac, B. Okac and J. Trtilek, Z. anal. Chem., **98**, 188 (1934) *28*

$CuCl_2$

$C_{18}H_{12}N_2$ **III-1072**

Cupric salts form colored complexes with 2:2′-biquinolyl:

$$(CuCl_2 + C_{18}H_{12}N_2 \rightarrow CuCl_2 \cdot C_{18}H_{12}N_2)$$

J. G. Breckenridge, R. W. J. Lewis and L. A. Quinck, Can. J. Res., **B17**, 258 (1939) *69*

$CuCl_2$

III-1073

CaC_2

Cupric chloride can be reduced to copper if it is heated with calcium carbide until the mixture is red hot. The reaction is often of explosive character.

$$CuCl_2 + CaC_2 \rightarrow Cu + CaCl_2 + 2C$$

F. v. Kügelgen, Z. Elektrochem., **7**, 559 (1901) *86*

$CuCl_2$

III-1074

Cu

Cuprous chloride may be prepared by dissolving cupric chloride, copper oxide, or copper carbonate in hydrochloric acid, adding strips of copper foil (in excess) and water, and heating for an hour.

$$CuCl_2 + Cu \rightarrow Cu_2Cl_2$$

R. C. Roark, J.A.O.A.C., **3**, 335 (1920) *42*

$CuCl_2$

III-1075

Cu

Cupric chloride is very corrosive to copper in a wide variety of concentrations according to the reaction.

$$Cu + CuCl_2 \rightarrow 2CuCl$$

E. E. Thum, Chem. & Met. Eng., **26**, 301 (1922) *44*

$CuCl_2$

III-1076

CuS

Copper chloride, (10%) in concentrated sodium chloride solution will remove H_2S, etc. from gas that is passed through the solution, in the form of free sulfur.

(a) $(H_2S + CuCl_2 \rightarrow CuS + 2HCl)$

(b) $CuS + CuCl_2 \rightarrow Cu_2Cl_2 + S$

(c) $Cu_2S + 2CuCl_2 \rightarrow 2Cu_2Cl_2 + S$

F. Fischer and P. Dilthey, Brenn. Chem., **7**, 300 (1926) *11*

$CuCl_2$

Cu_2S **III-1077**

Cuprous sulfide reacts with a cupric chloride solution, precipitating sulfur and leaving cuprous chloride in solution.

$$2CuCl_2 + Cu_2S \rightarrow \underline{S} + 4CuCl$$

A. Larsson, Sv. Kem. Tidsk., **4**, 85 (1892) *10*

$CuCl_2$

$FeCl_2$ **III-1078**

KCNS

$TiCl_3$

Copper may be determined volumetrically, in the presence of a sufficient quantity of ferrous iron (a) and an excess of KCNS (b) by the agency of a standard solution of titanous chloride (c). The copper salt is titrated until the red color of ferric thiocyanate just disappears (d).

(a) $$CuCl_2 + FeCl_2 \rightleftharpoons CuCl + FeCl_3$$

(b) $$FeCl_3 + 3KCNS \rightleftharpoons Fe(CNS)_3 + 3KCl$$

(c) $$CuCl_2 + KCNS + TiCl_3 \rightarrow CuCNS + KCl + TiCl_4$$

(d) $$Fe(CNS)_3 + HCl + TiCl_3 \rightarrow Fe(CNS)_2 + HCNS + TiCl_4$$

W. M. Thornton, Jr., J. Am. Chem. Soc., **44**, 998 (1922) *16*

$CuCl_2$

FeS **III-1079**

Ferrous sulfide reacts with a cupric chloride solution, precipitating sulfur and leaving cuprous chloride and ferrous chloride in solution.

$$2CuCl_2 + FeS \rightarrow \underline{S} + 2CuCl + FeCl_2$$

A. Larsson, Sv. Kem. Tidsk., **4**, 85 (1892) *10*

$CuCl_2$

H_2O **III-1080**

SO_2

If a solution of cupric chloride is saturated with sulfur dioxide gas, it is reduced to cuprous chloride.

$$2CuCl_2 + SO_2 + 2H_2O \rightarrow Cu_2Cl_2 + 2HCl + H_2SO_4$$

Greenwalt, J. Ind. Eng. Chem., **2**, 555 (1910) *22*

$CuCl_2$

H_3PO_2 **III-1081**

Cupric chloride is reduced to cuprous chloride by hypophosphorous acid.

$$6CuCl_2 + 2H_3PO_2 \rightarrow 6CuCl + (H_3PO_3 + 3HCl + POCl_3)$$

A. Sieverts, Z. anorg. Chem., **64**, 29 (1909)
Ref., J. L. Howe, J. Am. Chem. Soc., **33**, 177 (1911) *1*

$CuCl_2$

H_3PO_3 **III-1082**

Cupric chloride is reduced to cuprous chloride by phosphorous acid.

$$2CuCl_2 + H_3PO_3 + H_2O \rightarrow 2CuCl + (H_3PO_4 + 2HCl)$$

A. Sieverts, Z. anorg. Chem., **64**, 29 (1909)
Ref., J. L. Howe, J. Am. Chem. Soc., **33**, 177 (1911) *1*

$CuCl_2$

H_3PO_4 **III-1083**

Copper chloride and phosphoric acid heated together on a sand bath (up to 400°C) gives copper dimetaphosphate.

$$CuCl_2 + 2H_3PO_4 \rightarrow Cu(PO_3)_2 + 2HCl + 2H_2O$$

I. J. Moltkehansen, Trans. Am. El. Chem. Soc., **4**, 40 (1903) *8*

$CuCl_2$

H_3PO_4 **III-1084**

Copper chloride treated on a steam bath with concentrated phosphoric acid in slight excess forms copper orthophosphate.

$$3CuCl_2 + 2H_3PO_4 \rightarrow Cu_3(PO_4)_2 + 6HCl$$

I. J. Moltkehansen, Trans. Am. El. Chem. Soc., **4**, 40 (1903) *8*

$CuCl_2$

KCl **III-1085**

$BaCl_2$

$NaNO_2$

Copper chloride and potassium chloride dissolved in a solution of sodium nitrite will react with a solution of barium chloride in sodium nitrite, yielding crystals of green color. The same type reaction occurs by replacing potassium chloride with ammonium chloride.

(a) $CuCl_2 + 2KCl + BaCl_2 + 6NaNO_2 \rightarrow CuBaK_2 \cdot 6NO_2 + 6NaCl$

(b) $CuCl_2 + 2NH_4Cl + BaCl_2 + 6NaNO_2 \rightarrow CuBa(NH_4)_2 \cdot 6NO_2 + 6NO_2 + 6NaCl$

C. Przibylla, Z. anorg. Chem., **15**, 419 (1897) *28*

$CuCl_2$

KOH **III-1086**

A precipitate is formed from $N/5$ solutions of cupric chloride and potassium hydroxide at 85°C.

$$4CuCl_2 + 6KOH \rightarrow \underline{CuCl_2 \cdot (CuO)_3 \cdot 2H_2O} + 6KCl + H_2O$$

Miller and F. B. Kenrick, J. Phys. Chem., **7**, 259 (1903) *7*

$CuCl_2$

KSCN **III-1087**

$TiCl_2$

By reducing an acid solution of a cupric salt to which potassium thiocyanate has been added by means of titanium chloride, cuprous thiocyanate is precipitated quantitatively.

$$(CuCl_2 + KSCN + TiCl_2 \rightarrow \underline{CuSCN} + TiCl_3 + KCl)$$

v. P. Gerlinger, Z. angew. Chem., **19**, 520 (1906)
Ref., E. Knecht, Ber., **36**, 166, 1549 (1903)
Ref., Rhead, J. Chem. Soc. (London), **89**, 1491 (1906) *109*

$CuCl_2$

$K_2Hg(CNS)_4$ **III-1088**

A solution of cupric chloride will react with a solution of mercuric potassium thiocyanate yielding green colored crystals of cupric mercuric thiocyanate.

$$CuCl_2 + K_2Hg(CNS)_4 \rightarrow \underline{CuHg(CNS)_4} + 2KCl$$

A. Rosenheim and R. Cohn, Z. anorg. Chem., **27**, 280 (1901) *28*

$CuCl_2$("CuCl")

NH_3 **III-1089**

Cupric chloride forms four addition compounds with dry ammonia gas. Those more rich in ammonia are very unstable and are formed only upon cooling of the cupric chloride.

(a) $CuCl_2 + NH_3 \rightarrow CuCl_2 \cdot NH_3$

(b) $CuCl_2 + NH_3 \rightarrow CuCl_2 \cdot NH_3$

(c) $CuCl_2 + 2NH_3 \rightarrow CuCl_2 \cdot 2NH_3$

(d) $CuCl_2 + 3NH_3 \rightarrow CuCl_2 \cdot 3NH_3$

P. P. Deherain, Compt. rend., **55**, 809 (1862) *29*

$CuCl_2$

NH_3 **III-1090**

$(NH_4)_4Fe(CN)_6$

Cupric chloride dissolved in very concentrated ammonia will react with a solution of ammonium ferrocyanide yielding crystals of cupric ferrocyanide octammoniate of black color.

$$2CuCl_2 + 8NH_3 + (NH_4)_4Fe(CN)_6 \rightarrow \underline{Cu_2Fe(CN)_6 \cdot 8NH_3} + 4NH_4Cl$$

J. Messner, Z. anorg. Chem., **8**, 368 (1895) *28*

$CuCl_2$

NH_4Cl **III-1091**

Cupric chloride is reduced by ammonium chloride when heated together.

$$3CuCl_2 + NH_4Cl \rightarrow 3CuCl + 4HCl + N$$

P. Fireman, J. Am. Chem. Soc., **26**, 746 (1906) *1*

$CuCl_2$("CuCl")

NH_4Cl **III-1092**

Cupric chloride unites with ammonium chloride to form double salts.

(a) $CuCl_2 + 2NH_4Cl \rightarrow CuCl_2 \cdot 2NH_4Cl$

(b) $CuCl_2 + 3NH_4Cl \rightarrow CuCl_2 \cdot 3NH_4Cl$

P. P. Deherain, Compt. rend., **55**, 809 (1862) *29*

$CuCl_2$

NH_4Cl **III-1093**

$CaCl_2$

$NaNO_2$

17 grams of copper II chloride and 10.7 grams of ammonium chloride dissolved in a very small amount of water will react with a solution of 11.1 grams calcium chloride in 100 ml sodium nitrite solution, yielding crystals of green color.

$$CuCl_2 + 2NH_4Cl + CaCl_2 + 6NaNO_2 \rightarrow \underline{CuCa(NH_4)_2 \cdot 6NO_2} + 6NaCl$$

C. Przibylla, Z. anorg. Chem., **15**, 419 (1897) *28*

$CuCl_2$

$NCSCH_2 \cdot COOH$ **III-1094**

Copper chloride reacts with thiocyanic acetic acid, yielding a black precipitate.

(a) $2CuCl_2 + NCSCH_2COOH + 5H_2O \rightarrow \underline{CuSCH_2COOCuOH \cdot 5H_2O} + ?$

(b) $4CuCl_2 + 3NCSCH_2COOH + 8H_2O \rightarrow \underline{(CuSCH_2COO)_2Cu \cdot CuSCH_2COOCu \cdot OH \cdot 8H_2O} + ?$

I. V. Dubsky and V. Lindelar, Mikrochem., **24**, 264 (1938) *28*

$CuCl_2$

NaF **III-1095**

CuO

Sodium fluoride reacts with copper oxide and copper chloride in the presence of arsenious acid to form cupric-ortho-arsenite and copper fluoride.

$$3CuO + 3CuCl_2 + As_2O_3 + 2H_2O + 6NaF \rightarrow Cu_3(AsO_3)_2 \cdot 2H_2O + 3CuF_2 + 6NaCl$$

L. P. Curtin, Ind. Eng. Chem., **19**, 994 (1927) *23*

$CuCl_2$

$Na_2S_2O_3$ **III-1096**

Cupric chloride is reduced to cuprous by sodium thiosulfate, when the solution is neutral or weakly acid.

$$2CuCl_2 + 2Na_2S_2O_3 \rightarrow 2CuCl + 2NaCl + Na_2S_4O_6$$

R. Gans, Germ. Pat. 105061
Ref., Z. angew. Chem., **12**, 1082 (1899) *34*

$CuCl_2$

$Na_6P_6O_{18}$ **III-1097**

A saturated solution of cupric chloride will react with a solution of sodium hexametaphosphate yielding a precipitate of copper hexametaphosphate of blue color.

$$3CuCl_2 + Na_6P_6O_{18} \rightarrow \underline{Cu_3P_6O_{18}} + 6NaCl$$

H. Lüdert, Z. anorg. Chem., **5**, 15 (1894) *28*

$CuCl_2$

O_2 **III-1098**

Free chlorine may be produced by treating cupric chloride with oxygen.

$$2CuCl_2 + O_2 \rightarrow 2CuO + 2Cl_2$$

Wm. Odling, Chem. News, **23**, 211 (1871) *101*

$CuCl_2$

O_2 **III-1099**

Cupric chloride and brass in the presence of oxygen forms zinc chloride, cupric chloride, and cupric oxychloride.

(a) $CuCl_2 + (2Cu \cdot Zn) \rightarrow ZnCl_2 + 3Cu$

(b) $3Cu + 3CuCl_2 \rightarrow 6CuCl$

(c) $6CuCl + 3O + 4H_2O \rightarrow Cu_4(OH)_6Cl_2 \cdot H_2O + 2CuCl_2$

G. D. Bengough and R. May,
Ref., Can. Chem. & Met., **8**, 268 (1924) *15*

$CuCl_2$

O_2 **III-1100**

Heating cupric chloride in the presence of oxygen will give copper oxychloride and chlorine.

$$4CuCl_2 + O_2 \rightarrow 2CuO \cdot CuCl_2 + 2Cl_2$$

M. G. Levi and V. Bettoni, Gazz. chim. ital., **35**, 324 (1905) *21*

$CuCl_2$

O_2 **III-1101**

HCl

Copper chloride reacts with oxygen and hydrochloric acid to form first copper oxide and chlorine and then to reform copper chloride and water.

(a) $CuCl_2 + O \rightarrow CuO + Cl_2$

(b) $CuO + 2HCl \rightarrow CuCl_2 + H_2O$

Kopp, Gazz. chim. ital., **1**, 519 (1871) *21*

$CuCl_2$

Zn **III-1102**

Cupric chloride attacks metallic zinc according to the reaction.

$$CuCl_2 + Zn \rightarrow ZnCl_2 + Cu$$

E. E. Thum, Chem. & Met. Eng., **26**, 301 (1922) *44*

$CuCl_2$

III-1103

Δ

Pure chlorine is produced by heating cupric chloride.

$$CuCl_2 \rightarrow (Cu) + Cl_2$$

K. B. Krauskopf and G. K. Rollefson, J. Am. Chem. Soc., **56**, 2542 (1934) *1*

$CuCl_2$

III-1104

Δ

When certain chlorides and oxides are introduced into an ordinary Bunsen gas flame, dissociation occurs and the metal can be collected on a cold porcelain tube held in the flame. The process has been tried successfully with copper chloride, cadmium chloride, stannous chloride, mercuric chloride, silver nitrate, lead nitrate, bismuth nitrate, zinc chloride, arsenious oxide, antimony trichloride, molybdic oxide, tungstic oxide, phosphorus trichloride, sodium chloride, potassium chloride, and sulfur dioxide. The last six compounds decompose in a hydrogen-air or hydrogen-oxygen flame.

$$CuCl_2 \rightarrow Cu + Cl_2$$

W. D. Bancroft and H. B. Weiser, J. Phys. Chem., **18**, 213 (1914) *7*

$CuCl_2$

III-1105

Δ

To prepare pure chlorine, cupric chloride was heated.

$$2CuCl_2 \rightarrow Cl_2 + (2CuCl)$$

Meyer Wilderman, Trans. Roy. Soc. (London), **199**, 341 (1902) *105*

$CuCl_2$

Aminocaprylic acid **III-1106**

Addition of a hot solution of aminocaprylic acid to a hot solution of cupric chloride produces a precipitate of cupric aminocaprylate.

$$2C_7H_{14}NH_2COOH + CuCl_2 \rightarrow \underline{(C_7H_{14}NH_2COO)_2Cu} + 2HCl$$

E. Erlenmeyer and O. Sigel, Ann., **176**, 348 (1875) *20*

$CuCl_2 \cdot CuCl \cdot NH_3$

HCl **III-1107**

When hydrochloric acid acts on the cuprous-cupric chloride ammonia addition compound, cuprous chloride, ammonium chloride and cupric chloride-ammonium chloride addition compounds are formed.

$$2[CuCl \cdot CuCl_2 \cdot NH_3] + 2HCl \rightarrow 2CuCl \cdot NH_4Cl + 2CuCl_2 \cdot NH_4Cl$$

P. P. Deherain, Compt. rend., **55**, 809 (1862) *29*

$CuCl_2 \cdot CuO$

Δ **III-1108**

When cupric oxychloride is heated at 400°, it gives up the oxygen, leaving cuprous chloride.

$$2CuCl_2 \cdot CuO \rightarrow 4CuCl + O_2$$

A. Mallet, Compt. rend., **64**, 226 (1867) *29*

$CuCl_2 \cdot 2H_2O$

I_2 **III-1109**

SO_2

CH_3OH

With a methanol solution of Karl Fischer reagent, copper in the cupric form is reduced to cuprous copper.

$$CuCl_2 \cdot 2H_2O + \tfrac{3}{2}I_2 + 2SO_2 + 2CH_3OH \rightarrow CuI + 2HCl + 2HI + 2HSO_4CH_3$$

J. Mitchell, Jr., D. M. Smith, E. C. Ashby and W. M. D. Bryant, J. Am. Chem. Soc., **63**, 2928 (1941) *16*

$CuCl_2 \cdot (KCl)_2 \cdot 2H_2O$

$CuCl_2 \cdot 2H_2O$ **III-1110**

Cupric-potassium chloride dihydrate reacts with cupric chloride dihydrate when warmed above 56.2 degrees forming cupric-potassium chloride and water.

$$CuCl_2 \cdot (KCl)_2 \cdot 2H_2O + CuCl_2 \cdot 2H_2O \rightarrow 2CuCl_2 \cdot KCl + 4H_2O$$

J. G. C. Vriens, Rec. trav. Chim., **10**, 161 (1891) *39*

$CuCl_2 \cdot (KCl)_2 \cdot 2H_2O$

Fe **III-1111**

To analyze the carbon content of steel, a current of nitrogen is slowly passed through a solution of cupric potassium chloride into which are introduced fine turnings of the sample. The reaction of cupric chloride on the steel is allowed to progress, with frequent agitation, for approximately two hours at about 45°C. Copper first precipitates (a), then redissolves (b), after which the temperature is increased to boiling and the gases evolved are collected in appropriate absorbers.

(a) $Fe + CuCl_2 \rightarrow FeCl_2 + \underline{Cu}$

(b) $Cu + CuCl_2 \rightarrow Cu_2Cl_2$

E. Goutal, Compt. rend., **148**, 989–990 (1909) *38*

$CuCl_2 \cdot (KCl)_2 \cdot 2H_2O$

Δ **III-1112**

The double potassium cupric chloride dihydrate decomposes above 92.4 degrees forming potassium cupric chloride, potassium chloride, and water.

$$CuCl_2 \cdot 2KCl \cdot 2H_2O \leftrightarrows CuCl_2 \cdot KCl + KCl + 2H_2O$$

J. G. C. Vriens, Rec. trav. Chim., **10**, 161 (1891) *39*

$(CuCl_2)_2 \cdot NH_3$

HCl **III-1113**

Hydrochloric acid reacts with two of the cupric ammonia chlorides to form double chlorides.

(a) $2(CuCl_2) \cdot NH_3 + 2HCl \rightarrow 2CuCl_2 \cdot NH_4Cl$

(b) $CuCl_2 \cdot NH_3 + HCl \rightarrow CuCl_2 \cdot NH_4Cl$

P. P. Deherain, Compt. rend., **55**, 809 (1862) *29*

$CuCr_2O_7$

$Hg(CN)_2$ **III-1114**

Crystals of dark brown color are obtained when a solution of cupric dichromate reacts with a solution of mercuric cyanide.

$$CuCr_2O_7 + Hg(CN)_2 + 5H_2O \rightarrow \underline{CuCr_2O_7 \cdot Hg(CN)_2 \cdot 5H_2O}$$

G. Krüss and O. Unger, Z. anorg. Chem., **8**, 368 (1895) *28*

CuF_2

NH_3 **III-1115**

A solution of cupric fluoride will react with concentrated ammonia. The resulting liquid is shaken with a mixture of alcohol and ether yielding an oil like substance. By leaving to dry over potassium hydroxide in vacuo, after 4 weeks, blue colored crystals are formed.

$$CuF_2 + 4NH_3 + 5H_2O \rightarrow \underline{CuF_2 \cdot (NH_3)_4 \cdot 5H_2O}$$

E. Böhm, Z. anorg. Chem., **43**, 326 (1905) *28*

$CuFeS_2$

S_2Cl_2 **III-1116**

Chalcopyrite is decomposed by sulphur monochloride when the mixture is heated in a sealed tube.

$$2CuFeS_2 + 5S_2Cl_2 \rightarrow 2CuCl_2 + 2FeCl_3 + 7S_2$$

E. F. Smith, J. Am. Chem. Soc., **20**, 290 (1898) *1*

$Cu(HCO_2)_2$

Δ **III-1117**

When copper formate in water is heated to 170°, copper crystallizes out and Cu_2O, CO_2 and H_2 are formed.

$$3Cu(HCO_2)_2 + H_2O \rightarrow \underline{Cu} + Cu_2O + 6CO_2 + 4H_2$$

Riban,
Ref., A. Henninger, Ber., **9:2**, 1133 (1876) *26*

$Cu(H_2NSO_4)_2$

Δ **III-1118**

There are four different possibilities when copper hydroxyamidosulphate is decomposed at high temperatures.

(a) $$Cu(H_2NSO_4)_2 \rightarrow N_2O + 2H_2O + 2SO_2 + CuO$$

(b) $$3Cu(H_2NSO_4)_2 \rightarrow 2N_2O + 4H_2O + 2SO_2 + 2CuSO_4 + Cu(H_2NSO_3)_2$$

(c) $$3Cu(H_2NSO_4)_2 \rightarrow 2N_2 + 2H_2O + 2H_2SO_4 + 2CuSO_4 + Cu(H_2NSO_3)_2$$

(d) $$Cu(H_2NSO_4)_2 \rightarrow N_2 + 2H_2O + SO_2 + CuSO_4$$

E. Divers and T. Haga, J. Chem. Soc. (London), **77:2**, 980 (1900) *105*

$Cu(H_2O)_x^{++}$

III-1119

ε

In the solution of a hydrated copper salt, the mechanism of the electrode process may be represented by three reactions: (a) reduction of cupric ion to cuprous ion; (b) reduction of cuprous ion to metallic copper; (c) reduction of cupric ion to metallic copper.

(a) $Cu(H_2O)_x^{++} + e^- \rightarrow Cu(H_2O)_y^{+} + (x - y)H_2O$

(b) $Cu(H_2O)_y^{+} + e^- \rightarrow Cu + yH_2O$

(c) $Cu(H_2O)_x^{++} + 2e^- \rightarrow Cu + zH_2O$

Stewart B. Watkins and Henry G. Denham, J. Chem. Soc. (London), **115**:**2**, 1276 (1919) *48*

CuI

C_2H_2 **III-1120**

KI

By passing acetylene into a concentrated solution of cuprous iodide in potassium iodide, a yellow-orange precipitate is formed, which becomes red upon washing with a saturated solution of potassium iodide.

(a) $2CuI + C_2H_2 + KI \rightarrow (HC_2Cu_2I \cdot KI + HI)$

(b) $HC_2Cu_2I \cdot KI \rightarrow HC_2Cu_2I + KI$

Bertholet, Compt. rend., **62**, 457 (1866) *29*

CuI

$C_2H_5SCH_2COOH$ **III-1121**

Cuprous iodide and ethylthioglycolic acid are heated gently and benzol added. Small, colorless, lustrous crystals of anhydrous ethylthioglycolic acid cuprous iodide separate slowly.

$$CuI + C_2H_5SCH_2COOH \rightarrow (CuI) \cdot (C_2H_5SCH_2COOH)$$

L. Ramberg, Acta Univ. Lund. Med. Matem. Natur., N. S., Vol. **4**, No. 2, 13 (1908) *10*

CuI

$C_9H_7NCH_3I$ **III-1122**

Cuprous iodide and quinoline methiodide react to form the red double compound listed below.

$$CuI + C_9H_7NCH_3I \rightarrow C_9H_7NCH_3CuI_2$$

M. Kohn, Monatsh., **33**, 919 (1912) *4*

CuI

H_2S **III-1123**

Hydrogen sulfide when passed into an aqueous suspension of cuprous iodide forms hydriodic acid and cuprous sulfide.

$$2CuI + H_2S \rightarrow Cu_2S + 2HI$$

George Langbein, Ber., **7**, 766 (1874) *11*

CuI

I_2 **III-1124**

The reversible complex salt formation between cuprous iodide and iodine is studied.

$$2CuI + I_2 \rightleftharpoons 2CuI_2$$

K. Elbs, Z. Elektrochem., **23**, 147 (1917) *86*

CuI

MnO_2 **III-1125**

Cuprous iodide is heated with manganese dioxide.

$$2CuI + 3MnO_2 \rightarrow 2CuO + Mn_3O_4 + I_2$$

T. Schmidt, Chem. News, **37**, 56 (1878) *25*

CuI

RbCN **III-1126**

By saturating a solution of rubidium cyanide with cuprous iodide, crystals of rubidium cuprous cyanide are obtained.

$$3CuI + 5RbCN \rightarrow \underline{Rb_2Cu_3(CN)_5} + 3RbI$$

H. Grossmann and P. v. d. Forst, Z. anorg. Chem., **43**, 94 (1905) *28*

$Cu(IO_3)_2$

III-1127

NH_3

Copper iodate is dissolved in ammonia and gaseous ammonia is passed into this solution. Crystals of deep-blue color are formed.

$$Cu(IO_3)_2 + 4NH_3 + 2H_2O \rightarrow \underline{Cu(IO_3)_2 \cdot (NH_3)_4 \cdot 2H_2O}$$

F. Ephraim and A. Jansen, Ber., **48**, 50 (1915) *28*

$Cu(IO_3)_2 \cdot (NH_3)_4 \cdot 2H_2O$

III-1128

Δ

NH_3

When copper iodate tetrammoniate dihydrate is kept in a glass tubing at 110°, water distills off. On cooling in an ammonia atmosphere copper iodate pentammoniate is formed.

(a) $Cu(IO_3)_2 \cdot (NH_3)_4 \cdot 2H_2O \rightarrow Cu(IO_3)_2 \cdot 4NH_3 + 2H_2O$

(b) $Cu(IO_3)_2 \cdot 4NH_3 + NH_3 \rightarrow \underline{Cu(IO_3)_2 \cdot 5NH_3}$

F. Ephraim and A. Jansen, Ber., **48**, 50 (1915) *28*

CuI_2

III-1129

Decomp.

Cupric iodide decomposes spontaneously into cuprous iodide and free iodine.

$$2CuI_2 \rightarrow 2CuI + I_2$$

P. Klason, Sv. Kem. Tidsk., **31**, 211 (1919) *10*

CuI_2

III-1130

Δ

By heating cupric iodide, the result will be cuprous iodide and iodine.

$$2CuI_2 \rightarrow 2CuI + I_2$$

E. Oliveri Mandata, Gazz. chim. ital., **40**, 107 (1910) *21*

$CuKNaC_4O_6H_2$

H_2O **III-1131**

Hydrolysis of the complex compound formed in the neutralization of Fehling's solution gives colloidal copper hydroxide which is reduced to cuprous oxide by reducing sugars:

(a) $CuKNaC_4O_6H_2 + 2H_2O \rightarrow KNaC_4O_6H_4 + \underline{Cu(OH)_2}$

(b) $4Cu(OH)_2 \rightarrow 2Cu_2O + O_2 + 4H_2O$

J. L. Delsal, J. chim. phys., **35**, 314 (1938) *69*

$CuNH_2$

KNH_2 **III-1132**

Cuprous amide reacts with potassium amide in liquid ammonia to give a precipitate which dissolves in excess precipitant.

$$CuNH_2 + 2KNH_2 \rightarrow \underline{CuNK_2} + 2NH_3 \rightarrow (K_2[Cu(NH_2)_3])$$

E. C. Franklin, J. Phys. Chem., **23**, 40 (1919) *86*

$CuCO_3(NH_3)_4$

CO **III-1133**

NH_3

With occasional agitation, copper carbonate tetrammoniate solutions with a total copper content of 1 to 5% and not over 1% cupric copper in the presence of an excess of ammonia become colorless on the addition of CO (within an hour at room temperature and within less than 10 minutes at 60°).

$$2CuCO_3(NH_3)_4 + CO + 2H_2O - (4 - 2n)NH_3 \rightarrow Cu_2(NH_3)_{2n}CO_3 + 2(NH_4)_2CO_3$$

W. R. Hainsworth and E. V. Titus, J. Am. Chem. Soc., **43**, 8 (1921) *16*

$CuCO_3(NH_3)_4$

Cu **III-1134**

After cupric ammonium carbonate solution had been prepared by agitating, in air, ammonium carbonate and ammonium hydroxide, the air was turned off and the solution mixed until the cupric copper was reduced.

$$CuCO_3(NH_3)_4 + Cu - (4 - 2n)NH_3 \rightarrow (CuCO_3(NH_3)_n)_2$$

W. R. Hainsworth and E. V. Titus, J. Am. Chem. Soc., **43:1**, 1 (1921) *16*

$Cu(NH_3)Cl$

III-1135

O_2

When oxygen is passed thru a solution of cuprous ammonium chloride, there is formed a complex addition compound and cupric oxide which remains dissolved in the excess ammonia.

$$4Cu(NH_3)Cl + O_2 + 3H_2O \rightarrow (CuCl_2)_2 \cdot CuO \cdot (NH_3)_2 \cdot 3H_2O + CuO + 2NH_3$$

P. P. Deherain, Compt. rend., **55**, 809 (1862) *29*

$Cu(NO_2)_2$

III-1136

ε

The products formed by the electrolysis of copper nitrite solution may be explained by (a) the formation of a cupric anion $Cu(NO_2)_6^{----}$, (b) the decomposition of this ion, and (c) the slow decomposition of undissociated nitrous acid.

(a) $$Cu^{++} + 6NO_2^- \rightarrow Cu(NO_2)_6^{----}$$

(b) $$2Cu(NO_2)_6^{----} + 8H^+ + H_2O \rightarrow Cu(NO_2)_2 \cdot CuO + 10HNO_2$$

(c) $$3HNO_2 \rightarrow HNO_3 + 2NO + H_2O$$

F. Jeffery, Trans. Faraday Soc., **16**, 457, 709 (1920) *85*

$Cu(NO_2)_2$

III-1137

Δ

When a solution of cupric nitrite is evaporated under diminished pressure, nitric oxide is evolved and a residue of copper oxide and copper nitrate is left.

$$3Cu(NO_2)_2 \rightarrow Cu(NO_3)_2 + 2CuO + 4NO$$

P. C. Rây, J. Chem. Soc. (London), **91**:**2**, 1405 (1907) *102*
103

$CuNO_3$

K_2S **III-1138**

The titration of copper as sulfide can be done by the conductivity method in solutions containing at least 0.02 gr. of metal per litre and slightly acidified with acetic acid.

$$2CuNO_3 + K_2S \rightarrow \underline{Cu_2S} + 2KNO_3$$

P. Dutoit and G. von Weisse, J. Chim. phys., **9**, 608 (1911) *69*

$Cu(NO_3)_2$

Ag **III-1139**

ε

When a solution of copper nitrate is electrolyzed in a solution with a silver anode, a copper-silver alloy is deposited on the cathode.

$$2Cu(NO_3)_2 + 6Ag \rightarrow 2CuAg + 4AgNO_3$$

J. W. Richards, Trans. Am. El. Chem. Soc., **5**, 93 (1904) *8*

$Cu(NO_3)_2$

CH_3COONa **III-1140**

When hot solutions of copper nitrate react with sodium acetate, a blue-greenish precipitate of basic copper nitrate is obtained.

$$4Cu(NO_3)_2 + 6CH_3COONa + 3H_2O \rightarrow \underline{Cu(NO_3)_2 \cdot 3CuO} + 6NaNO_3 + 6CH_3COOH$$

W. Casselmann. Z. anal. Chem., **4**, 24 (1865) *28*

$Cu(NO_3)_2$

$C_9H_{12}ON_2S$ **III-1141**

Copper nitrate reacts with 5-cyclohexanone-2-thiohydantoine, yielding a black precipitate.

$$6Cu(NO_3)_2 + 5C_9H_{12}ON_2S \rightarrow \underline{(C_9H_{10}ON_2SCu)_5 \cdot Cu(NO_3)_2} + 10HNO_3$$

I. V. Dubsky, V. Lindelar and V. Cernak, Mikrochem., **25**, 124 (1938) *28*

$Cu(NO_3)_2$

Fe **III-1142**

By the addition of pure iron to a solution of a soluble salt of copper, copper may be obtained in its pure metallic state.

$$(Fe + Cu(NO_3)_2 \rightarrow Fe(NO_3)_2 + \underline{Cu})$$

C. Bischof, Chem. News., **8**, 133 (1863) *101*

$Cu(NO_3)_2$

HCl **III-1143**

Cupric chloride was obtained by evaporating a solution of cupric nitrate to a small volume, adding 5 cc of concentrated hydrochloric acid, and evaporating to dryness on a steam bath.

$$Cu(NO_3)_2 + 2HCl \rightarrow CuCl_2 + 2HNO_3$$

Bosee and Fehder, J. Am. Pharm. Assn., **29**, 141 (1940) *112*

$Cu(NO_3)_2$

H_2 **III-1144**

Hydrogen when passed thru a solution of copper nitrate reduces it to copper nitrite.

$$Cu(NO_3)_2 + 2H_2 \rightarrow Cu(NO_2)_2 + 2H_2O$$

R. Gerstl, Ber., **6**, 1424 (1873) *11*
Ref., W. J. Russell, J. Chem. Soc. (London), **27**, 11 (1874) *48*

$Cu(NO_3)_2$

H_2 **III-1145**

HNO_3

Nitrous acid was prepared by reducing cupric nitrate to cupric nitrite with hydrogen then is displacing it with nitric acid.

(a) $$Cu(NO_3)_2 + 2H_2 \rightarrow Cu(NO_2)_2 + (2H_2O)$$

(b) $$Cu(NO_2)_2 + 2HNO_3 \rightarrow 2HNO_2 + (Cu(NO_3)_2)$$

V. H. Veley, Trans. Roy. Soc. (London), **182**, 314 (1891) *105*

$Cu(NO_3)_2$

KI **III-1146**

Iodine is liberated when a slightly alkaline solution of cupric nitrate is treated with an excess of potassium iodide crystals.

$$2Cu(NO_3)_2 + 4KI \rightarrow 2CuI + 4KNO_3 + I_2$$

E. W. Rockwood, J. Am. Chem. Soc., **39**, 2746 (1917) *1*

$Cu(NO_3)_2$

KNH_2 **III-1147**

Black cuprous nitride is formed when the olive-green cuprous compound which is obtained by the reaction of potassium amide on copper nitrate is heated for a considerable time.

$$3Cu(NO_3)_2 + 6KNH_2 \rightarrow Cu_3N + 6KNO_3 + 4NH_3 + N$$

F. F. Fitzgerald, J. Am. Chem. Soc., **29**, 659 (1907) *1*

$Cu(NO_3)_2$

K_2CrO_4 **III-1148**

When a solution of copper nitrate is mixed with one of potassium chromate only the normal chromate is formed:

$$(K_2CrO_4 + Cu(NO_3)_2 \rightarrow \underline{CuCrO_4} + 2KNO_3)$$

P. E. Pelletier, L. Cloutier and P. E. Gagnon, Can. J. Res., **B16**, 37 (1938) *69*

$Cu(NO_3)_2$

K_2TeO_4 **III-1149**

When a solution of copper nitrate is treated with a solution of potassium, tellurate green copper tellurate is produced, which when boiled with water is converted into copper orthotellurate.

(a) $Cu(NO_3)_2 + K_2TeO_4 \rightarrow \underline{CuTeO_4} + (2KNO_3)$

(b) $3CuTeO_4 + 2H_2O \rightarrow \underline{Cu_3TeO_6} + (2H_2TeO_4)$

E. B. Hutchins, J. Am. Chem. Soc., **27**, 1181 (1905) *1*

$Cu(NO_3)_2$

NH_3 **III-1150**

Crystals of copper nitrate tetrammoniate are formed when ammonia gas is passed into a solution of copper nitrate.

$$Cu(NO_3)_2 + 4NH_3 \rightarrow \underline{Cu(NO_3)_2 \cdot 4NH_3}$$

F. F. Fitzgerald, J. Am. Chem. Soc., **29**, 657 (1907) *1*
Ref., Fritz Ephraim and Edward Bolle, Ber., **48**, 1770 (1916) *28*

$Cu(NO_3)_2$

Na_2CO_3 **III-1151**

Basic copper carbonate is produced by pouring a cold saturated solution of copper nitrate into an ice cold solution of sodium carbonate.

$$3Cu(NO_3)_2 + 3Na_2CO_3 + H_2O \rightarrow \underline{2CuCO_3 \cdot Cu(OH)_2} + (6NaNO_3 + CO_2)$$

A. C. Neish and J. W. Burns, Can. Chem. & Met., **5**, 73 (1921) *15*

$Cu(NO_3)_2$

Na_2S **III-1152**

Copper nitrate is transformed to copper sulfide by its reaction with sodium sulfide.

$$(Cu(NO_3)_2 + Na_2S \rightarrow \underline{CuS} + 2NaNO_3)$$

C. Kunsel, Chem. News, **8**, 38 (1863) *101*

$Cu(NO_3)_2$

$Pb(NO_3)_2$ **III-1153**

NH_4NO_3

$NaNO_2$

Black crystals are obtained when a solution of copper II nitrate and lead nitrate reacts with a solution of ammonium nitrate and sodium nitrite.

$$Cu(NO_3)_2 + Pb(NO_3)_2 + 2NH_4NO_3 + 6NaNO_2 \rightarrow \underline{CuPb(NH_4)_2 \cdot 6NO_2} + 6NaNO_3$$

C. Przibylla, Z. anorg. Chem., **15**, 419 (1897) *28*

$Cu(NO_3)_2$

ZnS **III-1154**

Copper nitrate reacts with zinc sulfide to produce solid copper sulfide.

$$(Cu(NO_3)_2 + ZnS \rightarrow \underline{CuS} + Zn(NO_3)_2)$$

C. Kunsel, Chem. News, **8**, 38 (1863) *101*

$Cu(NO_3)_2$

Δ **III-1155**

Electrolytic copper converted into nitrate was carefully ignited. It was completely converted to the oxide.

$$2Cu(NO_3)_2 \rightarrow 2CuO + (4NO_2 + O_2)$$

C. R. Wright and A. P. Luff, J. Chem. Soc. (London), **33**, 7 (1878) *81*

$Cu(NO_3)_2 \cdot 4NH_3$

Cu **III-1156**

The corresponding cuprous salt forms when dried cupric nitrate tetrammoniate is treated with copper foil and ammonia under pressure.

$$Cu(NO_3)_2 \cdot 4NH_3 + Cu \rightarrow 2[CuNO_3 \cdot 2NH_3]$$

W. H. Sloan, J. Am. Chem. Soc., **32**, 973 (1910) *1*

$Cu(NO_3)_2 \cdot 4NH_3$

KNH_2 **III-1157**

Cupric nitrate tetrammoniate reacts with potassium amide to form ammoniated cuprous nitride and this compound in turn reacts with an excess of potassium amide to form potassium ammonocuprite.

(a) $3Cu(NO_3)_2 \cdot 4NH_3 + 6KNH_2$
$\rightarrow Cu_3N \cdot nNH_3 + 6KNO_3 + (16 - n)NH_3 + N$

(b) $Cu_3N \cdot nNH_3 + 6KNH_2 + (5 - n)NH_3 \rightarrow 3CuNK_2 \cdot 3NH_3$

E. C. Franklin, J. Am. Chem. Soc., **34**, 1506 (1912) *1*

$Cu(NO_3)_2 \cdot 4NH_3$

K_2HgI_4 **III-1158**

Cupric nitrate tetrammoniate will react with a 10% solution of potassium mercuric iodide yielding a crystalline precipitate of green color.

$$Cu(NO_3)_2 \cdot 4NH_3 + 2K_2HgI_4 \rightarrow \underline{Cu(HgI_3)_2 \cdot 4NH_3} + 2KNO_3 + 2KI$$

A. Taurius, Z. anal. Chem., **97**, 29 (1934) *28*

$Cu(NO_3)_2 \cdot 4NH_3$

III-1159

NH_3

Copper nitrate hexammoniate is obtained when copper nitrate tetrammoniate is reacted upon with ammonia gas at low temperature.

$$Cu(NO_3)_2 \cdot 4NH_3 + 2NH_3 \rightarrow \underline{Cu(NO_3)_2 \cdot 6NH_3}$$

Fritz Ephraim and Edward Bolle, Ber., **48**, 1770 (1916) *28*

$CuNaC_4O_6H_3$

III-1160

H_2O

The addition compounds formed in the neutralization of Fehling's solution by sodium hydroxide are partly hydrolyzed:

(a) $CuNaC_4O_6H_3 + H_2O \rightleftharpoons CuC_4O_6H_4 + NaOH$

(b) $CuNaKC_4O_6H_2 + H_2O \rightleftharpoons CuKC_4O_6H_3 + NaOH$

J. L. Delsal, J. Chim. phys., **35**, 314 (1938) *69*

CuO

III-1161

C

SiO_2

Copper oxide, if fused with silica and carbon, yields a silicide:

$$3CuO + 5C + SiO_2 \rightarrow Cu_3Si + 5CO$$

K. Nischk, Z. Elektrochem., **29**, 383 (1923) *86*

CuO

III-1162

CH_3I

Methyl iodide vapor acts on heated cupric oxide to yield cuprous iodide without liberation of iodine.

$$CH_3I + CuO \rightarrow CuI + ?$$

H. G. Denham, J. Chem. Soc. (London), **111**, 29 (1917) *57*

CuO

CaC_2

III-1163

Cupric oxide is reduced to copper if it is heated with calcium carbide.

(a) $CuO + CaC_2 \rightarrow Cu + CaO + 2C$

(b) $4CuO + 2C \rightarrow 4Cu + 2CO_2$

(c) $5CuO + CaC_2 \rightarrow 5Cu + CaO + 2CO_2$

F. v. Kugelgen, Z. Elektrochem., **7**, 562 (1901) *86*

CuO

$CaCl_2$

III-1164

In converting the oxide of copper to the chloride, the ore is pulverized and brought into contact with calcium chloride. A stream of highly heated air is then passed through it.

$$2CaCl_2 + 4CuO \rightarrow 2CaO + 2Cu_2Cl_2 + O_2$$

Whelpley and Störer, (Chem. News, **1**, 106 (1867))? *101*

CuO

CaH_2

III-1165

When molecular proportions of copper oxide and calcium hydride are intimately mixed and fired by a fuse or taper, a very vigorous reaction occurs as follows:

$$2CuO + CaH_2 \rightarrow 2Cu + CaO + H_2O + \Delta$$

F. W. Perkin and L. Pratt, Trans. Faraday Soc., **3**, 182 (1907) *85*

CuO

Cu

NH_4Cl

III-1166

An addition product of cuprous chloride with ammonia is formed when ammonium chloride is boiled with cupric oxide and an excess of copper. This is often mixed with a cuprous-cupric chloride ammonia addition compound.

(a) $2CuO + 4Cu + 2NH_4Cl + 2H_2O$
$\rightarrow (CuCl)_2NH_3 \cdot 2H_2O + Cu_2O + NH_3(Cu_2Cl_2)$

(b) $6CuO + 4Cu + 6NH_4Cl + H_2O$
$\rightarrow 2(CuCl_2 \cdot CuCl \cdot NH_3 \cdot 2H_2O) + 4NH_3 + 3Cu_2O$

P. P. Deherain, Compt. rend., **55**, 808 (1862) *29*

CuO

Cu_2O **III-1167**

$CuO \cdot CuSO_4$

In an investigation of the catalytic activities of V_2O_5, CuO, and SiO_2 the monovariant equilibrium below is shown to determine the efficiency.

$$Cu_2O + CuO \cdot CuSO_4 \leftrightarrows 4CuO + SO_2.$$

B. Neumann, C. Kröger, and R. Iwanowski, Z. Elektrochem., **41**, 834 (1935) *86*

CuO

Fe **III-1168**

Cupric oxide reacts with iron when heated to form FeO.

(a) $$CuO + Fe \rightarrow FeO + Cu$$

(b) $$2CuO + Fe \rightarrow FeO + Cu_2O$$

K. Kautz, J. Am. Cer. Soc., **21**, 313 (1938) *16*

CuO

$FeCl_3$ **III-1169**

When cupric oxide and ferric chloride are heated together, ferric oxide and cupric chloride are formed.

$$3CuO + 2FeCl_3 \rightarrow Fe_2O_3 + 3CuCl_2$$

P. Klason, Sv. Kem. Tidsk., **29**, 192 (1917) *10*

CuO

FeO **III-1170**

At high temperatures cupric oxide reacts with FeO as follows.

$$2CuO + 3FeO \rightarrow Fe_3O_4 + Cu_2O$$

K. Kautz, J. Am. Cer. Soc., **21**, 313 (1938) *16*

CuO

$FeSO_4$ **III-1171**

Cupric oxide is dissolved by ferrous sulfate solutions to form copper sulfate, ferric oxide, sulfur trioxide and hydrogen.

$$CuO + 2FeSO_4 + H_2O \rightarrow CuSO_4 + (Fe_2O_3 + SO_3) + H_2$$

N. S. Keith, Trans. Am. El. Chem. Soc., **1**, 137 (1902) *8*

CuO

$Fe_2(SO_4)_3$ **III-1172**

Ferric sulfate solutions dissolve copper oxide to form copper sulfate and precipitate ferric oxide.

$$3CuO + Fe_2(SO_4)_3 \rightarrow 3CuSO_4 + Fe_2O_3$$

N. S. Keith, Trans. Am. El. Chem. Soc., **1**, 137 (1902) *8*

CuO

HCN **III-1173**

Curpic cyanide, which is very unstable, can be obtained in a state of purity by treating freshly precipitated cupric oxide with hydrocyanic acid.

$$CuO + 2HCN \rightarrow Cu(CN)_2 + H_2O \text{ ("CNCu")}$$

A. Lallemand, Compt. rend., **58**, 750 (1864) *29*

CuO

HCOOH **III-1174**

If copper oxide is boiled with formic acid and the solution filtered and cooled, fine blue crystals of copper formate are deposited.

$$CuO + 2HCOOH + 4H_2O \rightarrow Cu(HCOO)_2 \cdot 4H_2O + (H_2O)$$

F. W. Clark, Science, **1**, 67 (1883) *73*

CuO

HCl **III-1175**

Cupric oxide was dissolved in hydrochloric acid, forming cupric chloride and water.

$$CuO + 2HCl \rightarrow CuCl_2 + (H_2O)$$

H. Debus, Trans. Roy. Soc. (London), **173**, 540 (1882) *105*

CuO

HCl **III-1176**

Cupric oxide may be transformed into cupric chloride by treatment with hydrochloric acid.

$$CuO + 2HCl \rightarrow CuCl_2 + H_2O$$

Wm. Odling, Chem. News, **23**, 211 (1871) *101*
Ref., Greenwalt, J. Ind. Eng. Chem., **2**, 555 (1910) *22*

CuO

HI **III-1177**

Cupric oxide dissolves rapidly in liquid hydriodic acid liberating iodine.

(a) $$CuO + 2HI \rightarrow CuI_2 + H_2O$$

(b) $$2CuI_2 \rightarrow Cu_2I_2 + I_2$$

R. S. Norris and F. G. Cottrell, Am. Chem. J., **18**, 99 (1896) *1*

CuO

HNO_3 **III-1178**

Cupric oxide was dissolved in nitric acid, forming cupric nitrate and water.

$$CuO + 2HNO_3 \rightarrow Cu(NO_3)_2 + (H_2O)$$

H. Debus, Trans. Roy. Soc. (London), **173**, 539 (1882) *105*

CuO

H_2 **III-1179**

Copper may be formed by the reduction of pure cupric oxide by hydrogen.

$$(CuO + H_2 \rightarrow Cu + H_2O)$$

Berzelius, Pogg. Ann., **8**, 177
Ref., T. W. Richards, Am. Chem. J., **10**, 182 (1887) *1*

CuO

H_2 **III-1180**

Finely divided cupric oxide is ignited in an atmosphere of hydrogen to give pure metallic copper.

$$(CuO + H_2 \rightarrow Cu + H_2O)$$

W. Gibbs, Chem. News, **2**, 258 (1868) *101*
Ref., J. P. Cooke and T. W. Richards, Am. Chem. J., **10**, 90 (1888) *17*
Ref., J. L. St. John, J. Phys. Chem., **33**, 1439 (1929) *85*

CuO

H_2 **III-1181**

CO

Powdered copper is obtained when cupric oxide is reduced in a current of hydrogen or carbon monoxide.

(a) $$CuO + H_2 \rightarrow Cu + H_2O$$

(b) $$CuO + CO \rightarrow Cu + CO_2$$

P. Sabatier and J. B. Senderens, Ann. chim. phys. [7] (A), **7**, 399 (1896)
Ref., Tartar and Semon, J. Am. Chem. Soc., **43**, 494 (1921) *1*

CuO

H_2F_2 **III-1182**

Blue colored crystals of acid copper fluoride are obtained by leaving a concentrated solution of copper oxide in hydrofluoric acid in vacuo over sulfuric acid.

$$CuO + 7HF + 4H_2O \rightarrow \underline{CuF_2 \cdot (HF)_5 \cdot 5H_2O}$$

E. Böhm, Z. anorg. Chem., **43**, 326 (1905) *28*

CuO

H_2SO_4 **III-1183**

Sulfuric acid dissolves CuO to form copper sulfate.

$$CuO + H_2SO_4 \rightarrow CuSO_4 + H_2O$$

N. S. Keith, Trans. Am. El. Chem. Soc., **1**, 137 (1902) *8*

CuO

H_2SO_4 **III-1184**

Sulfuric acid reacts with copper oxide forming water and copper sulfate.

$$H_2SO_4 + CuO \rightarrow H_2O + CuSO_4$$

Greenwalt, J. Ind. Eng. Chem., **2**, 555 (1910) *22*

CuO

III-1185

MoO_3

HF

Copper fluoxymolybdate is obtained by dissolving copper oxide and molybdenum trioxide in hydrofluoric acid. After a few days blue crystals are formed.

$$CuO + 4HF + MoO_3 + 2H_2O \rightarrow \underline{CuF_2 \cdot MoO_2F_2 \cdot 4H_2O}$$

F. Memro, Z. anorg. Chem., **2**, 25 (1892) *28*

CuO

III-1186

NH_2OH

Hydroxylamine may be determined by use of Fehling's solution.

$$2NH_2OH + 4CuO \rightarrow N_2O + 2Cu_2O + (NO) + 3H_2O$$

W. Meyeringh, Ber., **10**, 940
Ref., Z. anal. Chem., **18**, 345 (1879) *1*
Ref., J. Am. Chem. Soc., **1**, 347 (1776)

CuO

III-1187

NH_2OH

When hydroxylamine is reacted with cupric oxide, cuprous oxide, nitrous oxide and water are formed.

$$2NH_2OH + 4CuO \rightarrow N_2O + 2Cu_2O + 3H_2O$$

H. O. Jones and F. W. Carpenter, J. Chem. Soc. (London), **83**, 1395 (1903) *102*

CuO

III-1188

NH_4COOH

HCOOH

A strong solution of ammonium formate is prepared. Another solution made by adding 30 gms. of NH_4COOH to 10 cc. water is prepared. Copper oxide is mixed with the second formate solution and enough of the first solution added to form a deep blue color. This solution is diluted with half its own volume of the first formate solution, and then with about 10 times its volume of alcohol. Formic acid is then added until the solution turns green and colorless crystals are deposited.

(a) $Cu_2O + 2HCOOH + 4NH_4OH \rightarrow Cu_2(OOCH)_2 \cdot 4NH_3 + 5H_2O$

(b) $Cu_2(OOCH)_2 \cdot 4NH_3 \rightarrow Cu_2(O_2CH)_2 + 4NH_3$

A. Angel, J. Chem. Soc. (London), **89:1**, 347 (1906) *103*

CuO

NH_4I **III-1189**

The reaction of copper oxide and ammonium iodide at 400°C. may be represented as follows.

$$2CuO + 4NH_4I \rightarrow 2CuI + 4NH_3 + I_2 + 2H_2O$$

Earle R. Caley and M. Gilbert Burford, Anal. Ed., **8**, 114 (1936) *44*

CuO

$(NH_4)_2S$ **III-1190**

Ammonium sulfide solution reacts with copper oxide to form cuprous sulfide, ammonia, water and sulfur.

$$2CuO + 2(NH_4)_2S \rightarrow Cu_2S + 4NH_3 + 2H_2O + S$$

Karl Heumann, Ber., **6**, 752 (1873) *11*
Ref., Gazz. chim. ital., **6**, 203 (1876) *21*

CuO

$(NH_4)_2S_x$ **III-1191**

Cupric oxide is added to yellow ammonium sulfide. It reacts to form cuprous sulfide.

$$2CuO + 2(NH_4)_2S_x \rightarrow Cu_2S + 4NH_3 + 2H_2O + S_x$$

K. Heumann, Ann., **173**, 31 (1874) *20*

CuO

$(NH_4)_2Sx$ **III-1192**

Copper oxide in contact with ammonium polysulfide (deep yellow) forms a pink double compound of cuprous sulfide and ammonium hexasulfide.

(a) $2CuO + (NH_4)_2Sx \rightarrow Cu_2S + (NH_4)_2Sx - 1 + (O_2)$

(b) $(Cu_2S + (NH_4)_2S_6 \rightarrow Cu_2S \cdot (NH_4)_2S_6)$

E. Priwoznik, Ber., **6**, 1293 (1873) *11*

CuO

III-1193

$SOCl_2$

Cupric chloride is formed when cupric oxide is heated at 200° for several hours with thionyl chloride in a sealed tube.

$$CuO + SOCl_2 \rightarrow CuCl_2 + SO_2$$

H. B. North and A. M. Hageman, J. Am. Chem. Soc., **35**, 356 (1913) *1*

CuO

III-1194

SO_2

Sulfur dioxide dissolved in absolute alcohol reacts with cupric oxide, producing a green powder insoluble in water.

$$4CuO + SO_2 + 7H_2O \rightarrow \underline{(CuO)_4 \cdot SO_2 \cdot 7H_2O}$$

Millon and Commaille, Compt. rend., **57**, 822 (1863) *29*

CuO

III-1195

SO_2

Cupric oxide, when heated in a stream of sulfur dioxide, reacts to form cuprous oxide and cupric sulfate. When the reaction products are heated longer at red heat, sulfur trioxide is evolved.

(a) $$3CuO + SO_2 \rightarrow Cu_2O + CuSO_4$$

(b) $$2CuSO_4 + SO_2 \rightarrow Cu_2O + 3SO_3$$

D. L. Hammick, J. Chem. Soc. (London), **111**, 384 (1917) *57*

CuO

III-1196

SO_2

O_2

When cupric oxide is heated in the presence of sulfur dioxide and air, cupric sulfate is formed.

$$2CuO + 2SO_2 + O_2 \rightarrow 2CuSO_4$$

P. Klason, Sv. Kem. Tidsk., **29**, 192 (1917) *10*

CuO

Zn **III-1197**

HCl

When an intimate mixture consisting of 14.2 parts of cupric oxide and 7 parts of zinc powder are added in small portions to concentrated hydrochloric acid, cuprous chloride is formed.

$$2CuO + Zn + 4HCl \rightarrow 2CuCl + ZnCl_2 + 2H_2O$$

Karl Heumann, Ber., **7**, 721 (1874) *11*

CuO

Δ **III-1198**

When heated, cupric oxide decomposes into cuprous oxide and oxygen. If oxygen is removed, the reaction proceeds to completion from left to right.

$$4CuO \rightleftharpoons 2Cu_2O + O_2$$

K. Kautz, J. Am. Cer. Soc., **21**, 313 (1938) *16*

CuO

Δ **III-1199**

Cupric oxide is transformed into cuprous oxide by heating at 1000° in an inert atmosphere:

$$4CuO \rightarrow 2Cu_2O + O_2$$

C. Stora, J. Chim. phys., **29**, 168 (1932) *69*

CuO

K_2CO_3 **III-1200**

Electrolytic copper converted into nitrate was boiled with freshly fused potash and invert sugar and the precipitated red cuprous oxide washed with distilled water. The product was 99.7% pure.

(a) $$Cu + 4HNO_3 \rightarrow Cu(NO_3)_2 + 2NO_2 + 2H_2O$$

(b) $$2Cu(NO_3)_2 + 2K_2CO_3 \rightarrow \underline{Cu_2O} + 4KNO_3 + 2CO_2 + \cdot O$$

C. R. Wright and A. P. Luff, J. Chem. Soc. (London), **33**, 7 (1878) *81*

CuO

Levulinic acid **III-1201**

A hot aqueous solution of levulinic acid is saturated with cupric oxide, filtered and concentrated. The precipitated copper levulinate is washed with alcohol and recrystallized from 91% alcohol.

$$2C_4H_7OCOOH + CuO \rightarrow (C_4H_7OCOO)_2Cu + H_2O$$

A. Grote, E. Kehrer and B. Tollens, Ann., **206**, 218 (1881) *20*

CuOH

$Fe_2(SO_4)_3$ **III-1202**

H_2SO_4

Cuprous hydroxide reduces ferric sulfate in the presence of sulfuric acid.

$$2CuOH + Fe_2(SO_4)_3 + H_2SO_4 \rightarrow 2CuSO_4 + 2FeSO_4 + 2H_2O$$

Meade, J. Am. Chem. Soc., **20**, 610 (1898)
Ref., J. Am. Chem. Soc., **27**, 1235 (1905) *1*

CuOH

$KMnO_4$ **III-1203**

Cuprous hydroxide is oxidized to cupric hydroxide by potassium permanganate in alkaline solution.

$$8CuOH + 2KMnO_4 + 5H_2O \rightarrow 2KOH + 8Cu(OH)_2 + Mn_2O_3$$

S. W. Parr, J. Am. Chem. Soc., **22**, 687 (1900) *1*

CuOH

NH_4SCN **III-1204**

Dissolve the precipitated cuprous hydroxide in a very small amount of ammonia. By adding a concentrated solution of ammonium thiocyanate, a white powder is obtained.

$$2Cu(OH) + 2NH_4SCN \rightarrow \underline{Cu_2(SCN)_2 \cdot 2NH_3} + 2H_2O$$

T. W. Richards and B. S. Merigold, Z. anorg. Chem., **17**, 245 (1898) *28*

$Cu(OH)_2$

$C_4H_7BrCOOH$ **III-1205**

A concentrated aqueous solution of alphabromoisovaleric acid is digested with excess cupric hydroxide, the solution filtered and the residue digested with absolute alcohol. Filter and reduce alcohol to small volume by distillation. Crystals of cupric alphabromoisovalerinate form.

$$2C_5H_8O_2Br + Cu(OH)_2 \rightarrow (C_5H_7O_2Br)_2Cu + 2H_2O$$

B. Schleicher, Ann., **267**, 117 (1892) *20*

$Cu(OH)_2$

$C_6H_4OH \cdot COONa$ **III-1206**

Green crystals of copper sodium salicylate are formed when copper hydroxide is dissolved in sodium salicylate.

$$Cu(OH)_2 + 2C_6H_4 \cdot OH \cdot COONa \rightarrow \underline{CuO_2(C_6H_4COONa)_2} + 2H_2O$$

I. Wolff, Z. anal. Chem., **40**, 459 (1901) *28*

$Cu(OH)_2$

HN_3 **III-1207**

NH_4OH

Dark green transparent crystals are formed when the green precipitate formed by treating copper hydroxide with triazoic acid is washed with strong ammonium hydroxide.

(a) $(2Cu(OH)_2 + 4HN_3 \rightarrow 2CuN_6(5H_2O + O))$

(b) $CuN_6 + 2NH_4OH \rightarrow CuN_6 \cdot 2NH_3 + (2H_2O)$

L. M. Dennis and H. Isham, J. Am. Chem. Soc., **29**, 216 (1907) *1*

$Cu(OH)_2$

H_2F_2 **III-1208**

Copper fluoride is formed when cupric hydroxide is dissolved in hydrofluoric acid.

$$Cu(OH)_2 + H_2F_2 \rightarrow CuF_2 + 2H_2O$$

F. H. Edmister and H. C. Cooper, J. Am. Chem. Soc., **42**, 2424 (1920) *1*

$Cu(OH)_2$

III-1209

H_2O_2

Finely dispersed copper hydroxide is oxidized with 15%–30% hydrogen peroxide at 0°. Brown colored copper peroxide is obtained, provided the solution was neutral.

$$Cu(OH)_2 + H_2O_2 \rightarrow CuO_2 \cdot H_2O + H_2O$$

L. Moser, Z. anorg. Chem., **54**, 121 (1907) *28*

$Cu(OH)_2$

III-1210

$H_2Pt(C_2O_4)_2$

Cupric hydroxide (or carbonate) treated with platino-oxalic acid yields small, rod-like, dark grayish-brown crystals of hexa-hydrated cupric platino-oxalate.

$$Cu(OH)_2 + H_2Pt(C_2O_4)_2 + 4H_2O \rightarrow CuC_2O_4 \cdot PtC_2O_4 \cdot 6H_2O$$

H. G. Söderbaum, K. Sven. Vet. Ak. Handl. Övers., **42**, No. 10, 35 (1885) *10*

$Cu(OH)_2$

III-1211

H_2SeO_3

Copper hydroxide dissolves in selenous acid. At ordinary temperature grayish blue, di-hydrated copper hydrogen selenite is obtained. This, when heated under water, becomes green and changes to the normal salt.

(a) $Cu(OH)_2 + 2H_2SeO_3 \rightarrow CuSeO_3 \cdot H_2SeO_3 \cdot H_2O + H_2O$

(b) $CuSeO_3 \cdot H_2SeO_3 \cdot (H_2O) \rightarrow \underline{CuSeO_3} + H_2SeO_3 + H_2O$

L. F. Nilson, K. Sven. Vet. Ak. Handl. Övers., **31**, No. 1, 42 (1874) *10*

$Cu(OH)_2$

III-1212

KBr

Copper hydroxide reacts with a solution of potassium bromide yielding oxygen.

(a) $3Cu(OH)_2 + 2KBr \rightarrow Cu_2Br_2 \cdot Cu(OH)_2 \cdot H_2O + K_2O_2 \cdot H_2O$

(b) $K_2O_2 \cdot H_2O \rightarrow 2KOH + O$

W. Spring and M. Lucion, Z. anorg. Chem., **2**, 218 (1892) *28*

$Cu(OH)_2$

$2NH_2OH \cdot H_2SO_4$ **III-1213**

A light green solution is formed when cupric hydroxide is dissolved in hydroxylamine hydrosulfate.

$$2Cu(OH)_2 + (NH_2OH)_2 \cdot H_2SO_4 \rightarrow Cu_2OSO_4 \cdot 2NH_2OH + 3H_2O$$

M. Adams and E. Overman, J. Am. Chem. Soc., **31**, 639 (1909) *1*

$Cu(OH)_2$

NH_3 **III-1214**

HCOOH

Formic acid is quickly changed to carbon dioxide when heated to 150° in a sealed tube in the presence of ammoniacal copper (hydroxide).

(a) $HCOOH + 2Cu(OH)_2 + 2NH_3 \rightarrow Cu_2O + 2H_2O + H_2CO_3 \cdot (NH_3)_2$

(b) $CO_3H_2(NH_3)_2 + Cu_2O + O \rightarrow (CuCO_3 \cdot 2NH_3) + Cu(OH)_2$

(c) $CuCO_3(NH_3)_2 \rightarrow CO_2 + CuO + 2NH_3$

P. Cazeneuve, J. Am. Chem. Soc., **1**, 490 (1876) *1*

$Cu(OH)_2$

NH_3 **III-1215**

$(NH_4)_2SO_4$

Cupric hydroxide reacts with ammonium sulfate and ammonia to form water and copper-sulfate tetrammoniate.

$$Cu(OH)_2 + 2NH_3 + (NH_4)_2SO_4 \rightarrow CuSO_4 \cdot 4NH_3 + 2H_2O$$

H. M. Dawson, J. Chem. Soc. (London), **95**:1, 374 (1909) *103*

$Cu(OH)_2$

$(NH_4)_2F_2$ **III-1216**

Blue crystals are formed by the reaction of copper hydroxide and ammonium fluoride. Copper hydroxide dissolves in hot ammonium fluoride and the excess water has to be evaporated.

$$Cu(OH)_2 + 2(NH_4)_2F_2 \rightarrow \underline{CuF_2 \cdot (NH_4)_2F_2} + 2H_2O + 2NH_3$$

H. V. Helmholt, Z. anorg. Chem., **3**, 115 (1893) *28*

$Cu(OH)_2$

III-1217

$(NH_4)_2SO_4$

Ammonium sulfate reacts with cupric hydroxide to form ammonia, water, and a basic copper sulfate salt.

$$2Cu(OH)_2 + (NH_4)_2SO_4 \rightarrow Cu_2(OH)_2SO_4 + 2NH_3 + 2H_2O$$

H. M. Dawson, J. Chem. Soc. (London), **95**:1, 374 (1909) *103*

$Cu(OH)_2$

III-1218

NaOH

(NH_4OH)

Crystalline cupric hydroxide, when treated with concentrated alkali solutions at room temperature or shaken with aqueous ammonia at 25°, is converted to cupric oxide.

$$Cu(OH)_2 + NaOH \rightarrow CuO + H_2O + NaOH$$

A. J. Allmand, J. Chem. Soc. (London), **97**, 603 (1910) *57*

$Cu(OH)_2$

III-1219

$Na_2C_{20}H_6Br_4O_5$

A suspension of copper hydroxide on standing in contact with an eosin suspension may produce (1) an amorphous lake of undefined composition, or (2) a crystalline product corresponding to the copper salt of an acid eosin, or (3) a mixture of (1) and (2):

$$Cu(OH)_2 + Na_2C_{20}H_6Br_4O_5 \rightarrow \underline{CuC_{20}H_6Br_4O_5} + 2NaOH$$

H. N. Gilbert, J. Phys. Chem., **18**, 586 (1914) *7*

$Cu(OH)_2$

III-1220

p-Aminophenylalanine

A not too dilute, hot aqueous solution of p-aminophenylalanine is treated with copper hydroxide, the solution filtered and concentrated. Crystals of cupric p-aminophenylalaninate separate.

$$2C_8H_{11}N_2COOH + Cu(OH)_2 \rightarrow Cu(C_8H_{11}N_2COO)_2 + 2H_2O$$

E. Erlenmeyer and A. Lipp, Ann., **219**, 223 (1883) *20*

$Cu(OH)_2$

l-Amino-n-valeric acid **III-1221**

A dilute aqueous solution of l-amino-n-valeric acid is heated with an excess of copper hydroxide, filtered and concentrated on the water-bath. Crystals of cupric l-amino-n-valerinate separate.

$$2C_4H_8NH_2COOH + Cu(OH)_2 \rightarrow \underline{Cu(C_4H_8NH_2COO)_2} + 2H_2O$$

A. Lipp, Ann., **211**, 362 (1882) *20*

$Cu(OH)_2$

Glutamic acid **III-1222**

A boiling saturated aqueous solution of glutamic acid is treated with cupric hydroxide as long as it dissolves. The solution is diluted with hot water until all of the crystals are dissolved, then the solution is filtered and cooled. Cupric glutamate crystallizes with two and one half H_2O.

$$C_3H_5NH_2(COOH)_2 + Cu(OH)_2 \rightarrow CuC_3H_5NH_2(COO)_2 + 2H_2O$$

F. Hofmeister, Ann., **189**, 22 (1877) *20*

$Cu(OH)_2$

Glycerol **III-1223**

In a determination of glycerol an addition compd. is formed in dil. alcoholic soln, then the addition compd. is determined in aliquot portion by reaction in acid soln, with potassium iodide, the liberated iodine being determined by titration.

$$Cu(OH)_2 + NaOH + CH_2OHCHOHCH_2OH \rightarrow CuNa(C_3H_5O_3)\cdot 3HOH$$

Bertram and Rutgers, Rec. trav. Chim., **57**, 681 (1938)
Ref., Oil and Soap, **16**, 1, 19, (1939) *111*

$Cu(OH)_2$

Leucine **III-1224**

A boiling saturated aqueous solution of leucine (alpha amino isocaproic acid) is treated with cupric hydroxide as long as it dissolves. The solution is diluted with hot water until all of the crystals dissolve and then filtered. Crystals of cupric leucinate deposit as the solution cools. The salt crystallizes with two H_2O.

$$2C_6H_{13}NO_2 + Cu(OH)_2 \rightarrow Cu(C_6H_{12}NO_2)_2 + 2H_2O$$

F. Hofmeister, Ann., **189**, 16 (1877) *20*

$Cu(OH)_2$

III-1225

Tyrosine

A hot aqueous solution of tyrosine is treated with cupric hydroxide, filtered and concentrated over sulfuric acid. Crystals of cupric tyrosinate are formed.

$$2C_8H_{10}NOCOOH + Cu(OH)_2 \rightarrow Cu(C_8H_{10}NOCOO)_2 + 2H_2O$$

E. Erlenmeyer and A. Lipp, Ann., **219**, 176 (1883) *20*

$CuO_2 \cdot H_2O$

III-1226

HCl

With dilute hydrochloric acid, monohydrated copper dioxide gives cupric chloride, hydrogen peroxide, water, and a small amount of oxygen.

(a) $CuO_2 \cdot H_2O + 2HCl \rightarrow CuCl_2 + H_2O + H_2O_2$

(b) $2CuO_2 \cdot H_2O + 4HCl \rightarrow 2CuCl_2 + O_2 + 4H_2O$

C. Weltzien, Compt. rend., **63**, 520 (1866) *29*

$Cu(PO_3)_2$

III-1227

$(NH_4)_2S$

When copper metaphosphate is treated with ammonium sulfide with a small excess of ammonium hydroxide, ammonium metaphosphate and copper sulfide are formed.

$$Cu(PO_3)_2 + (NH_4)_2S \rightarrow 2NH_4(PO_3) + CuS$$

I. J. Moltkehansen, Trans. Am. El. Chem. Soc., **4**, 40 (1903) *8*

CuP_2O_6

III-1228

H_2S

A fine powder of copper metaphosphate is treated with a solution of hydrogen sulfide for 16 hours. The filtrate of the copper sulfide contains metaphosphoric acid.

$$CuP_2O_6 + H_2S \rightarrow \underline{CuS} + 2HPO_3$$

F. Warschauer, Z. anorg. Chem., **36**, 137 (1903) *28*

$CuPt(CN)_4$

CaO **III-1229**

Calcium platinocyanide is prepared by boiling a suspension of calcium oxide and copper platinocyanide.

$$CuPt(CN)_4 + CaO \rightarrow CaPt(CN)_4 + (CuO)$$

L. A. Levy, J. Chem. Soc. (London), **93**, 1448 (1908) *57*

CuS

$AgNO_3$ **III-1230**

Cupric sulfide is immediately decomposed by the addition of a solution of silver nitrate.

$$CuS + 2AgNO_3 \rightarrow Ag_2S + Cu(NO_3)_2$$

A. Rössing, Z. anorg. Chem., **25**, 407 (1900) *28*

CuS

CaO **III-1231**

C

Copper sulfide mineral (or impurity) may be desulfurized by treating in the fused state with lime and excess carbon. An equilibrium exists in the distribution of S between metal and flux.

$$CuS + CaO + C \rightarrow Cu + CaS + CO$$

B. Bogitch, Trans. Faraday Soc., **20**, 139 (1924) *85*

CuS

HNO_3 **III-1232**

Cupric sulfide was dissolved in nitric acid, forming cupric nitrate and hydrogen sulfide. The hydrogen sulfide formed will be oxidized by the acid and the reaction is written for oxidization to sulfuric acid; nitrogen dioxide and water are also made.

(a) $$CuS + 2HNO_3 \rightarrow Cu(NO_3)_2 + (H_2S)$$

(b) $$H_2S + 8HNO_3 \rightarrow (8NO_2 + 4H_2O + H_2SO_4)$$

H. Debus, Trans. Roy. Soc. (London), **173**, 539 (1882) *105*

CuS

III-1233

HNO_3

Copper sulfide was dissolved by pouring several portions of boiling 25% nitric acid through the retentive filter upon which it had been collected after precipitation by hydrogen sulfide. The filtrate containing copper nitrate was analyzed micro-colorimetrically.

$$CuS + 2HNO_3 \rightarrow Cu(NO_3)_2 + H_2S$$

Bosee and Fehder, J. Am. Pharm. Assm., **29**, 141 (1940) *112*

CuS

III-1234

H_2

Hydrogen reduces cupric sulfide to cuprous sulfide at red heat.

$$2CuS + H_2 \rightarrow Cu_2S + (H_2S)$$

Karl Heumann, Ber., **6**, 749 (1873) *11*

CuS

III-1235

Na_2CO_3

Sodium thiosulfate is a product when covellite is heated with 10% sodium carbonate in a sealed tube filled with carbon dioxide for 22 hours at 180°.

(a) $8CuS + 3Na_2CO_3 \rightarrow 4Cu_2S + 2Na_2S + Na_2S_2O_3 + 3CO_2$

(b) $2CuS + Na_2S \rightarrow Cu_2S + Na_2S_2$

H. N. Stokes, J. Am. Chem. Soc., **29**, 313 (1907) *1*

CuS

III-1236

$SOCl_2$

Copper chloride is produced when copper sulfide is heated with thionyl chloride at 150° in a sealed tube.

$$CuS + 2SOCl_2 \rightarrow CuCl_2 + SO_2 + S_2Cl_2$$

H. B. North and C. B. Conover, J. Am. Chem. Soc., **37**, 2489 (1915) *1*

CuS

H_2 **III-1237**

ε

Copper sulfide is dissolved in a sulfuric acid solution and electrolyzed to form metallic copper and sulfur which in turn reacts with the hydrogen formed to yield hydrogen sulfide.

(a) $Cu_2S \rightarrow 2Cu + S$

(b) $S + H_2 \rightarrow H_2S$

A. T. Weightman, Trans. Am. El. Chem. Soc., **2**, 75 (1903) *8*

CuS

Δ **III-1238**

Cupric sulfide is reduced to cuprous sulfide when heated in a vacuum. In like manner, SnS_2 is reduced to SnS and Ag_2S reduced to 2Ag.

$$2CuS \rightarrow Cu_2S + S$$

F. Damm and F. Kraft, Ber., **40**, 4775 (1907)
Ref., W. Robertson, J. Chem. Soc. (London), **94:2**, 39 (1908) *57*

CuSCN

KIO_3 **III-1239**

HCl

Cuprous thiocyanate is oxidized and iodine monochloride is formed when a solution of cuprous thiocyanate is titrated with potassium iodate in the presence of a large excess of hydrochloric acid.

$$4CuSCN + 7KIO_3 + 14HCl \rightarrow 4CuSO_4 + 7KCl + 7ICl + 4HCN + 5H_2O$$

G. S. Jamieson, L. H. Levy and H. L. Wells, J. Am. Chem. Soc., **30**, 760 (1908) *1*

CuSCN

NH_3 **III-1240**

If ammonia gas is conducted over powdered cuprous thiocyanate, a black powder is formed.

$$2CuSCN + 10NH_3 \rightarrow \underline{2Cu(SCN)\cdot 5NH_3}$$

T. W. Richards and B. S. Merigold, Z. anorg. Chem., **17**, 245 (1898) *28*

CuSCN

NaOH **III-1241**

Cuprous thiocyanate is converted into cuprous hydroxide by caustic soda.

$$CuSCN + NaOH \rightarrow CuOH + NaCNS$$

W. E. Garrigues, J. Am. Chem. Soc., **19**, 941 (1897) *1*

$Cu(SCN)_2$

H_2O **III-1242**

Cupric thiocyanate is decomposed by water with the formation of cuprous thiocyanate, thiocyanic acid, hydrocyanic acid and sulfuric acid.

$$6Cu(SCN)_2 + 4H_2O \rightarrow 6Cu(SCN) + 5HSCN + HCN + H_2SO_4$$

E. Söderbäck, Sv. Kem. Tidsk., **21**, 116 (1909) *10*

$Cu(SCSOC_2H_5)_2$

Δ **III-1243**

Upon being ignited, copper xanthate produces the following probable reaction.

$$(Cu(SCSOC_2H_5)_2 \rightarrow CuS + COS + CS \cdot OC_2H_5 \cdot SC_2H_5)$$

Weiss, J. Ind. Eng. Chem., **1**, 605 (1909) *22*

$CuSO_4$

$AgNO_3$ **III-1244**

$Na_2S_2O_3$

Crystals of cuprous-sodium silver thiosulfate separate when an ammoniacal solution of sodium thiosulfate is added to a mixture of copper and silver salts.

$$(4AgNO_3 + 2CuSO_4) + 9Na_2S_2O_3 + 8NH_4OH$$
$$\rightarrow \underline{Cu_2S_2O_3 \cdot 2Ag_2S_2O_3 \cdot 5Na_2S_2O_3 \cdot 6NH_3} + (4NaNO_3$$
$$+ 2Na_2SO_4 + 7H_2O + (NH_4)_2S_2O_3) + O$$

O. L. Shinn, J. Am. Chem. Soc., **26**, 949 (1904) *1*

$CuSO_4$

$Ba(C_2H_5 \cdot SO_4)_2$ **III-1245**

Solutions of copper sulfate and barium ethyl sulfate are mixed, the barium sulfate filtered off, and the filtrate yields pretty, blue, somewhat efflorescent prisms and tablets of tetra-hydrated cupric ethyl sulfate.

$$CuSO_4 + Ba(C_2H_5 \cdot SO_4)_2 + 4H_2O \rightarrow \underline{BaSO_4} + Cu(C_2H_5 \cdot SO_4)_2 \cdot 4(H_2O)$$

J. E. Alèn, K. Sven. Vet. Ak. Handl. Övers., **37**, No. 8, 35 (1880) *10*

$CuSO_4$

$Ba(C_{10}H_5Cl(NHC_2H_3O) \cdot SO_3)_2$ **III-1246**

When solutions of copper sulfate and of the barium salt of chloracetnaphthylamine sulfonic acid are mixed and the barium sulfate filtered off, the filtrate yields the copper salt of chloracetnaphthylamine sulfonic acid as thin, almost colorless, easily soluble needle crystals.

$$CuSO_4 + Ba(C_{10}H_5Cl(NHC_2H_3O) \cdot SO_3)_2 \rightarrow \underline{BaSO_4} + Cu(C_{10}H_5Cl(NHC_2H_3O) \cdot SO_3)_2$$

P. Hellström, K. Sven. Vet. Ak. Handl. Bih., **16**, II, No. 3, 67 (1890) *10*

$CuSO_4$

$Ba(C_{10}H_5ClNH_2 \cdot SO_3)_2$ **III-1247**

When solutions of copper sulfate and of the barium salt of chloroamidonaphthalenesulfonic acid are mixed and the barium sulfate filtered off, the filtrate yields light green, easily soluble copper salt of chloroamidonaphthalenesulfonic acid.

$$CuSO_4 + Ba(C_{10}H_5ClNH_2 \cdot SO_3)_2 \rightarrow \underline{BaSO_4} + Cu(C_{10}H_5ClNH_2 \cdot SO_3)_2$$

P. Hellström, K. Sven. Vet. Ak. Handl. Bih., **16**, II, No. 3, 57 (1890) *10*

$CuSO_4$

III-1248

$Ba(C_{10}H_5(NO_2)_2 \cdot SO_3)_2$

When solutions of copper sulfate and of the barium salt of dinitronaphthalenesulfonic acid are mixed and the barium sulfate filtered off, the filtrate yields the tetra-hydrated copper salt of dinitronaphthalenesulfonic acid as well-formed, greenish-yellow needles, which are occasionally tablet-shaped, but which form short, thick needles on slow evaporation of the liquid.

(a) $CuSO_4 + Ba(C_{10}H_5(NO_2)_2 \cdot SO_3)_2 \rightarrow \underline{BaSO_4} + Cu(C_{10}H_5 \cdot (NO_2)_2 \cdot SO_3)_2$

(b) $Cu(C_{10}H_5(NO_2)_2 \cdot SO_3)_2 + 4H_2O \rightarrow Cu(C_{10}H_5(NO_2)_2 \cdot SO_3)_2 \cdot 4H_2O$

P. Hellström, K. Sven. Vet. Ak. Handl. Bih., **16**, II, No. 3, 41–44 (1890) *10*

$CuSO_4$

III-1249

$Ba(NO_2)_2$

Cupric nitrite may be prepared by the double decomposition of cupric sulfate and barium nitrite.

$$(CuSO_4 + Ba(NO_2)_2 \rightarrow Cu(NO_2)_2 + \underline{BaSO_4})$$

W. Hampe, Ann., **125**, 345 (1863)
Ref., Ray, J. Chem. Soc. (London), **91**, 1405 (1907) *109*

$CuSO_4$

III-1250

$CHCl_3$

Anhydrous cupric chloride is produced when anhydrous cupric sulfate is heated in a current of dry carbon dioxide and chloroform at 280–300°.

$$3CuSO_4 + 2CHCl_3 \rightarrow 3CuCl_2 + (H_2O + 3SO_3 + 2CO)$$

Conduché, Compt. rend., **158**, 1180 (1914)
Ref., J. L. Howe, J. Am. Chem. Soc., **37**, 548 (1915) *1*

$CuSO_4$

CH_3COONa **III-1251**

HCOONa

When a hot solution of copper sulfate reacts with sodium acetate, a blue-greenish precipitate of basic copper sulfate is obtained. The same type reaction occurs with sodium formate, sodium propionate, and sodium valerate.

(a) $4CuSO_4 + 6CH_3COONa + 3H_2O \rightarrow \underline{CuSO_4 \cdot 3CuO} + 3Na_2SO_4 + 6CH_3COOH$

(b) $4CuSO_4 + 6HCOONa + 3H_2O \rightarrow \underline{CuSO_4 \cdot 3CuO} + 3Na_2SO_4 + 6HCOOH$

(c) $4CuSO_4 + 6CH_3CH_2COONa + 3H_2O \rightarrow \underline{CuSO_4 \cdot 3CuO} + 3Na_2SO_4 + 6CH_3CH_2COOH$

(d) $4CuSO_4 + 6C_5H_9O_2Na + 3H_2O \rightarrow \underline{CuSO_4 \cdot 3CuO} + 3Na_2SO_4 + 6C_5H_{10}O_2$

W. Casselmann, Z. anal. Chem., **4**, 24 (1865) *28*

$CuSO_4$

C_5H_5N **III-1252**

$K_2Cr_2O_7$

Copper sulfate will react with solutions of potassium dichromate and pyridine yielding green colored, complex copper pyridine dichromate.

$$CuSO_4 + K_2Cr_2O_7 + 4C_5H_5N \rightarrow \underline{Cu(C_5H_5N)_4 \cdot Cr_2O_7} + K_2SO_4$$

P. Spacu, Z. anal. Chem., **115**, 424 (1938–39) *28*

$CuSO_4$

$C_6H_4OHCOOH$ **III-1253**

A solution of 3.5 g. salicylic acid in 20 ml. sodium hydroxide (1 g. NaOH) is added to a solution of 1.6 g. crystallized copper sulfate in 10 ml. of water. Olive green colored leaflets are formed.

$$CuSO_4 + 2C_6H_4OHCOOH + 4NaOH \rightarrow (C_7H_4O_3Na)_2Cu \cdot 4H_2O + Na_2SO_4$$

H. Ley and O. Erler, Z. anorg. Chem., **56**, 401 (1908) *28*

$CuSO_4$

III-1254

$C_6H_5COONH_4$

C_5H_5N

Copper sulfate will react with ammonium benzoate and pyridine yielding a crystalline precipitate of blue color.

$$CuSO_4 + 2C_5H_5N + 2C_6H_5COONH_4 \rightarrow \underline{(C_5H_5N)_2(C_6H_5COO)_2Cu} + (NH_4)_2SO_4$$

T. Dick and A. Radulescu, Z. anal. Chem., **111**, 394 (1937) *28*

$CuSO_4$

III-1255

$C_6H_5COONH_4$

$C_6H_{12}N_4$

Copper sulfate will react with ammonium benzoate and hexamethylene tetramine, yielding a crystalline precipitate of blue color.

$$3CuSO_4 + 6C_6H_5COONH_4 + C_6H_{12}N_4 \rightarrow \underline{Cu_3(C_6H_5COO)_6C_6H_{12}N_4} + 3(NH_4)_2SO_4$$

J. Dick, Z. anal. Chem., **111**, 260 (1937) *28*

$CuSO_4$

III-1256

$C_7H_5NS_2$

Copper sulfate will react with an alcoholic solution of mercapto benzothiazol yielding a precipitate.

$$CuSO_4 + 2C_7H_5NS_2 \rightarrow \underline{Cu(C_7H_4NS_2)_2} + H_2SO_4$$

G. Spacu and M. Kuras, Z. anal. Chem., **102**, 26 (1935) *28*

$CuSO_4$

III-1257

$C_7H_6O_2NNa$

A solution of copper sulfate will react with the sodium salt of anthranilic acid, yielding a green precipitate.

$$CuSO_4 + 2C_7H_6O_2NNa \rightarrow \underline{Cu(C_7H_6O_2N)_2} + Na_2SO_4$$

H. Funk and M. Ditt, Z. anal. Chem., **93**, 245 (1933) *28*

$CuSO_4$

$C_{10}H_6O_4$ **III-1258**

$C_{10}H_7O_3N$

Copper sulfate reacts with a solution of naphtazarine in alcohol, yielding a dark purple powder. The same type reaction occurs with naphtazarine imine.

(a) $CuSO_4 + C_{10}H_6O_4 + H_2O \rightarrow \underline{C_{10}H_4O_4Cu \cdot H_2O} + H_2SO_4$

(b) $CuSO_4 + C_{10}H_7O_3N + 9H_2O$
$\rightarrow \underline{C_{10}H_5O_3N \cdot Cu \cdot 9H_2O} + H_2SO_4$

I. V. Dubsky, A. Langer and E. Wagner, Mikrochem., **22**, 108 (1937) *28*

$CuSO_4$

$C_{10}H_7NO_2$ **III-1259**

Copper sulfate will react with a solution of quanaldinic acid yielding a crystalline green precipitate.

$$CuSO_4 + 2C_{10}H_7NO_2 + H_2O \rightarrow \underline{(C_{10}H_6NO_2)_2Cu \cdot H_2O} + H_2SO_4$$

P. Ray and M. K. Bose, Z. anal. Chem., **95**, 404 (1933) *28*

$CuSO_4$

$C_{11}H_{14}O_7$ **III-1260**

Neutral water solutions of copper salts treated with an alcoholic solution of the ethyl ester of acetondioxalic acid give a practically insoluble precipitate which contains 19.88% of copper.

$$C_{11}H_{14}O_7 + CuSO_4 \rightarrow \underline{CuC_{11}H_{12}O_7} + H_2SO_4$$

A. Jílek and J. Lukas, Chem. List., **19**, 275 (1925) *5*

$CuSO_4$

$C_{13}Cl_4H_5OCOONa$ **III-1261**

A hot aqueous solution of sodium-benzoyl 3,4,5,6-tetrachlorobenzoate is treated with an aqueous solution of copper sulfate and the precipitate washed with water and dried. The compound is copper-1-benzoyl 3,4,5,6 tetrachlorobenzoate and it crystallizes with two H_2O.

$$2C_{13}H_5Cl_4OCOONa + CuSO_4 \rightarrow (C_{13}H_5Cl_4OCOO)_2Cu + Na_2SO_4$$

G. Kircher, Ann., **238**, 340 (1887) *20*

$CuSO_4$

III-1262

$C_{14}H_8O_5N$

$C_{30}H_{18}O_{10}N_2$

Copper sulfate reacts with quinalizarine imine and quinalizarine ethylene diamine, yielding dark purple powders.

(a) $CuSO_4 + C_{14}H_8O_5N + 7H_2O \rightarrow \underline{C_{14}H_6O_5NCu \cdot 7H_2O} + H_2SO_4$

(b) $CuSO_4 + C_{30}H_{18}O_{10}N_2 + 10H_2O \rightarrow C_{30}H_{16}O_{10}N_2Cu \cdot 10H_2O + H_2SO_4$

I. V. Dubsky, A. Langer and E. Wagner, Mikrochem., **22**, 108 (1937) *28*

$CuSO_4$

III-1263

CaO

H_2O

When lime water is added to copper sulfate solution, $20CuO \cdot 4SO_3 \cdot CaSO_4$ is precipitated at the point at which the copper in the solution ceases to react with ferrocyanide (a). When the solution becomes alkaline, $30CuO \cdot 3SO_3 \cdot 4CaSO_4$ is precipitated (b). Different precipitates are formed up to the point at which the copper continues to react with iron (c), and when 50 to 500 moles CaO are added for each mol $CaSO_4$, (d).

(a) $20CuSO_4 + 4CaO + 15H_2O \rightarrow \underline{20CuO \cdot 4SO_3 \cdot 3CaO \cdot CaSO_4} + (15H_2SO_4)$

(b) $30CuSO_4 + 27CaO \rightarrow \underline{30CuO \cdot 3SO_3 \cdot 4CaSO_4} + (23CaSO_4)$

(c) $10CuSO_4 + 4CaO + 8H_2O \rightarrow \underline{10CuO \cdot SO_3 \cdot 3CaO \cdot CaSO_4} + (8H_2SO_4)$

(d) $CuSO_4 + 4CaO \rightarrow \underline{CuO \cdot 3CaO} + (CaSO_4)$

S. U. Pickering, J. Chem. Soc. (London), **97**, 1855 (1910) *57*

$CuSO_4$

III-1264

$Cu(H_2PO_2)_2$

When copper hypophosphite reacts with cupric sulfate solution at 70°C, a greenish liquid is formed and then yellow cuprous hydride precipitates and then turns brown.

$$Cu(H_2PO_2)_2 + CuSO_4 \rightarrow \underline{Cu_2H_2} + 2HPO_3 + SO_2$$

Å. G. Ekstrand, Sv. Kem. Tidsk., **10**, 29 (1898) *10*

$CuSO_4$

Fe **III-1265**

If metallic iron is placed in a copper sulfate solution the iron replaces the copper ions in solution precipitating metallic copper.

$$CuSO_4 + Fe \rightarrow FeSO_4 + Cu$$

C. E. Williams and A. E. Anderson, J. Ind. Eng. Chem., **14**, 1058 (1922) *22*

$CuSO_4$

$FeSO_4$ **III-1266**

ε

A solution containing 39 g. of copper, 14.4 g. iron (Fe^{++}), 96 g. sulfuric acid per liter is electrolyzed. Copper is deposited on the cathode, while the sulfate anion oxidizes ferrous to the ferric sulfate.

$$2CuSO_4 + 4FeSO_4 \rightarrow 2Cu + 2Fe_2(SO_4)_3$$

E. Cohen, Z. Elektrochem., **1**, 53 (1894) *86*

$CuSO_4$

$FeSO_4$ **III-1267**

ε

Copper is deposited on the cathode when an acidified copper sulfate solution containing ferrous sulfate is electrolyzed.

$$2CuSO_4 + 4FeSO_4 \rightarrow 2Cu + 2Fe_2(SO_4)_3$$

N. S. Keith, Trans. Am. Electrochem. Soc., **1**, 136 (1902) *8*

$CuSO_4$

$Fe_2(SO_4)_3$ **III-1268**

Fe

When iron is added to an acidified $CuSO_4$ solution containing ferric sulfate, metallic copper is precipitated.

$$CuSO_4 + Fe_2(SO_4)_3 + 2Fe \rightarrow Cu + 4FeSO_4$$

N. S. Keith, Trans. Am. Electrochem. Soc., **1**, 131 (1902) *8*

$CuSO_4$

III-1269

$HC_6H_{11}O_2$

Caproic acid may be converted to the copper salt by neutralizing with potassium hydroxide and then adding copper sulfate in the correct molecular proportion. The copper derivative forms a fine blue powder when it is dried in air, but when heated to 100–125°, it suddenly turns green.

$$2HC_6H_{11}O_2 + CuSO_4 \rightarrow \underline{Cu(C_6H_{11}O_2)_2} + H_2SO_4$$

J. L. Sammis, J. Phys. Chem., **10**, 593 (1906) *7*

$CuSO_4$

III-1270

HCl

At 450°C, sulfuric acid is quantitatively displaced from copper sulfate by the action of dry hydrogen chloride gas.

$$CuSO_4 + 2HCl \rightarrow CuCl_2 + H_2SO_4$$

R. A. Beebe and D. B. Summers, J. Am. Chem. Soc., **50**, 20 (1928) *16*

$CuSO_4$

III-1271

HCl

ε

When a continuous current is passed through an aqueous series of electrolytes which does not contain a common ion a chemical reaction occurs. In the series HCl-$CuSO_4$-HCl the cupric sulfate solution is discolored at the left-hand contact while the color is intensified at the right-hand by the formation of cupric chloride.

$$CuSO_4 + 2HCl\ (+\ \varepsilon) \rightarrow CuCl_2 + H_2SO_4$$

M. Chanoz, Compt. rend., **148**, 986 (1909) *38*

$CuSO_4$

NaOH **III-1272**

H_2

Metallic copper and cuprous oxide precipitate when a cuprous salt is reduced by a hydrogen atmosphere. The existence of a tetra copper oxide (Cu_4O) is denied.

(a) $$2CuSO_4 + H_2 \rightarrow Cu_2SO_4 + H_2SO_4$$

(b) $$Cu_2SO_4 + NaOH \rightarrow Cu_2O + NaHSO_4$$

(c) $$Cu_2SO_4 + H_2 \rightarrow 2Cu + H_2SO_4$$

M. J. Murray, J. Phys. Chem., **35**, 1024 (1931) *86*

$CuSO_4$

H_2O **III-1273**

By continued boiling of a water solution of cupric sulfate, a basic sulfate is obtained.

$$6CuSO_4 + 9H_2O \rightarrow (CuO)_6(SO_3)_2 \cdot 5H_2O + 4H_2SO_4$$

S. U. Pickering, Chem. News, **47**, 181 (1883)
Ref., C. F. M., Science, **2**, 19 (1883) *73*

$CuSO_4$

H_2O **III-1274**

A green-blue precipitate of $3CuO \cdot SO_3$ is obtained by boiling copper sulfate solution or by treating copper sulfate and a little water in a sealed tube at 200°.

$$3CuSO_4 + 2H_2O \rightarrow \underline{(CuO)_3SO_3} + (2H_2SO_4)$$

S. U. Pickering, Chem. News, **47**, 181 (1883)
Ref., Shenstone, J. Chem. Soc. (London), **47**, 375 (1885)
Ref., S. U. Pickering, ibid., **97**, 1855 (1910) *57*

$CuSO_4$

III-1275

H_2O_2

The rate of decomposition of hydrogen peroxide is greatly accelerated by the presence of some copper ions.

(a) $CuSO_4 + H_2O_2 \rightleftarrows CuO_2 + H_2SO_4$

(b) $CuO_2 + H_2O \rightleftarrows CuO_2 \cdot H_2O$

(c) $2CuO_2 \cdot H_2O \rightleftarrows \underline{2Cu(OH)_2} + O_2$

(d) $Cu(OH)_2 + H_2SO_4 \rightleftarrows CuSO_4 + 2H_2O$

H. W. Rudel and M. M. Haring, Ind. Eng. Chem., **22**, 1235 (1930) *24*

$CuSO_4$

III-1276

H_2S

Copper sulfide precipitated at room temperature from cupric sulfate is practically pure cupric sulfide. When aging with H_2S, or on precipitation from boiling solution, some reduction (5%) to cuprous sulfide occurs.

(a) $CuSO_4 + H_2S \rightarrow CuS + H_2SO_4$

(b) $2CuS \rightarrow Cu_2S + S$

I. M. Kolthoff and E. Pearson, J. Phys. Chem., **36**, 642 (1932) *85*

$CuSO_4$

III-1277

H_2SO_3

KI

If a solution of copper sulfate is saturated with sulfurous acid and then treated with an iodine salt, cuprous iodide is immediately precipitated.

$$2CuSO_4 + 2KI + SO_2 + 2H_2O \rightarrow \underline{Cu_2I_2} + K_2SO_4 + 2H_2SO_4$$

B. Lean and W. H. Whatmough, J. Chem. Soc. (London), **73**, 149 (1898) *103*

$CuSO_4$

III-1278

H_2SO_4

Fe

The addition of iron to an acidified $CuSO_4$ solution precipitates copper.

$$CuSO_4 + H_2SO_4 + 2Fe \rightarrow \underline{Cu} + 2FeSO_4 + H_2$$

N. S. Keith, Trans. Am. El. Chem. Soc., **1**, 131 (1902) *8*

$CuSO_4$

H_3PO_2 **III-1279**

Cupric hydride is prepared when copper sulfate is treated with hypophosphorous acid.

$$CuSO_4 + 2H_3PO_2 + 2H_2O \rightarrow CuH_2 + (H_2SO_4 + 2H_3PO_3)$$

E. J. Bartlett, N. F. Rice and W. H. Merrill, Am. Chem. J., **19**, 50 (1897) *1*

$CuSO_4$

H_3PO_3 **III-1280**

Phosphorous acid reduces copper sulfate to metallic copper.

$$CuSO_4 + H_3PO_3 \rightarrow \underline{Cu} + (H_3PO_4 + SO_2 + O)$$

Sieverts, Z. anorg. Chem., **64**, 29 (1909)
Ref., J. L. Howe, J. Am. Chem. Soc., **33**, 177 (1911) *1*

$CuSO_4$

KBrO **III-1281**

Copper sulfate reacts with alkali-free potassium hypobromite, to give a green flocculent deposit of a basic sulfate. This changes into the blue copper hydroxide if alkali is added, and finally becomes black copper oxide.

$$3CuSO_4 + 4KBrO \rightarrow \underline{SO_3 \cdot 3CuO} + 2K_2SO_4 + 2Br_2 + O_2$$

H. Pauli, Z. Elektrochem., **3**, 477 (1897) *86*

$CuSO_4$

KCN **III-1282**

Copper sulfate and excess potassium cyanide react to form a complex.

$$2(CuSO_4 + 3KCN) \rightarrow 2(CuCN \cdot KCN) + C_2N_2 + 2K_2SO_4$$

F. Spitzer, Z. Elektrochem., **11**, 346 (1905) *86*

$CuSO_4$

III-1283

KCN

NH_3

A deep blue solution is formed when potassium cyanide is added to a solution of copper sulfate until the precipitate formed just dissolves, and ammonia added to the resulting solution.

(a) $CuSO_4 + 2KCN \rightarrow (Cu(CN)_2 + K_2SO_4)$

(b) $2Cu(CN)_2 + 6NH_3 + 2HOH \rightarrow Cu(OH)_2 \cdot 4NH_3 + (NH_4)_2Cu(CN)_4$

Beringer and Hutchin, Eng. Min. J., **62**, 390
Ref., J. Am. Chem. Soc., **19**, 28 (1897) *1*

$CuSO_4$

III-1284

KOH

KCNS

Cuprous thiocyanate is precipitated when a solution of copper sulfate is treated with potassium thiocyanate. This salt is then boiled with a solution of caustic potash and the resulting hydrated cuprous oxide reduces ferric chloride in the presence of hydrochloric acid.

(a) $2CuSO_4 + 2KCNS \rightarrow \underline{2CuCNS} + (K_2SO_4 + SO_2 + O_2)$

(b) $CuCNS + KOH \rightarrow Cu(OH) + KCNS$

(c) $Cu_2O + 2FeCl_3 + 2HCl \rightarrow 2CuCl_2 + 2FeCl_2 + H_2O$

R. K. Meade, J. Am. Chem. Soc., **20**, 610 (1898) *1*

$CuSO_4$

III-1285

KCNS

C_5H_5N

Copper sulfate will react with potassium thiocyanate and pyridine yielding a green precipitate which is insoluble in water.

$$CuSO_4 + 2KCNS + 2C_5H_5N \rightarrow \underline{(Cu(C_5H_5N)_2)(SCN)_2} + K_2SO_4$$

G. Spacu, Z. anal. Chem., **67**, 27 (1925–26) *28*

$CuSO_4$

KCNS **III-1286**

$(NH_2(CH_3)C_6H_3)_2$

Copper sulfate will react with potassium thiocyanate and a 2% solution of tolidine in alcohol, yielding a blue precipitate, insoluble in water.

$$CuSO_4 + 2KCNS + (NH_2(CH_3)C_6H_3)_2 \rightarrow K_2SO_4 + \underline{(Cu(NH_2 \cdot CH_3 \cdot C_6H_3)_2) \cdot (SCN)_2}$$

G. Spacu, Z. anal. Chem., **67**, 30 (1925–26) *28*

$CuSO_4$

KCNS **III-1287**

Pyridine

Copper sulfate solution was neutralized to phenolphthalein. To the solution was added in succession, 10 cc. chloroform, 1 cc. glacial acetic acid, 1 cc. of 10% potassium thiocyanate solution and 15 drops of pyridine and water. The copper thiocyanate-pyridine complex dissolved in the chloroform and was estimated colorimetrically from the depth of its green color.

$$CuSO_4 + 2KCNS + 2C_5H_5N \rightarrow Cu(SCN)_2 \cdot (C_5H_5N)_2 + K_2SO_4$$

Bosee and Fehder, J. Am. Pharm. Assn., **29**, 141 (1940) *112*

$CuSO_4$

$KC_{16}H_{31}O_2$ **III-1288**

Copper palmitate precipitates when cupric sulfate is added to an aqueous solution of potassium palmitate (made by adding potassium hydroxide, in chemically equivalent amount, to a solution of palmitic acid). The copper palmitate may be recovered as a blue powder by washing the precipitate and then drying at 100°.

$$CuSO_4 + 2KC_{16}H_{31}O_2 \rightarrow \underline{Cu(C_{16}H_{31}O_2)_2} + K_2SO_4$$

J. L. Sammis, J. Phys. Chem., **10**, 593 (1906) *7*

$CuSO_4$

$KH_2Co_3(CN)_{11} \cdot H_2O$ **III-1289**

Cupric cobalto cobalticyanide is formed when solutions of monopotassium cobaltocobalticyanide and cupric sulfate are mixed.

$$3CuSO_4 + 2KH_2Co_3(CN)_{11} \cdot H_2O + 2H_2O \rightarrow \underline{Cu_3(Co_3(CN)_{11})_2 \cdot 4H_2O} + (K_2SO_4 + 2H_2SO_4)$$

C. L. Jackson and A. M. Comey, Am. Chem. J., **19**, 275 (1897) *1*

$CuSO_4$

KI **III-1290**

When a solution of cupric ion is treated with an excess of potassium iodide, the following reaction takes place.

$$2CuSO_4 + 4KI \rightarrow 2CuI + I_2 + 2K_2SO_4$$

Bartholow Park, Anal. Ed., **3**, 77 (1931) *44*

$CuSO_4$

KI **III-1291**

Copper sulfate and potassium iodide solutions react to give potassium sulfate, copper iodide and free iodine.

$$2CuSO_4 + 4KI \rightarrow 2K_2SO_4 + 2CuI + I_2$$

J. W. Walker and M. V. Dover, J. Chem. Soc. (London), **87**, 1584 (1905) *102*
Ref., E. O. Mandata, Gazz. chim. ital., **40**, 107 (1910) *21*

$CuSO_4$

KI **III-1292**

Cupric sulfate and potassium iodide react to give cuprous iodide, iodine, and potassium sulfate.

$$CuSO_4 + 2KI \rightarrow CuI + I + K_2SO_4$$

H. W. Foote, J. Am. Chem. Soc., **60**, 1349 (1938) *16*
3

$CuSO_4$

KI **III-1293**

$NH_2C_6H_4C_6H_4NH_2$

Copper sulfate will react with potassium iodide and a 1% solution of benzidine in alcohol yielding a blue precipitate, insoluble in water.

$$CuSO_4 + 2KI + NH_2C_6H_4C_6H_4NH_2 \rightarrow \underline{K_2SO_4(Cu(NH_2C_6H_4 \cdot C_6H_4NH_2)(I_2)}$$

G. Spacu, Z. anal. Chem., **67**, 32 (1925–26) *28*

$CuSO_4$

KNO_3 **III-1294**

ε

When a continuous current is passed through an aqueous series of electrolytes which does not contain a common ion, a chemical reaction occurs. In the series KNO_3-$CuSO_4$-KNO_3 the cupric sulfate solution is discolored at the left-hand contact while the color is intensified at the right-hand by formation of cupric nitrate.

$$CuSO_4 + 2KNO_3 + \varepsilon \rightarrow Cu(NO_3)_2 + K_2SO_4$$

M. Chanoz, Compt. rend., **148**, 986 (1909) *38*

$CuSO_4$

KOH **III-1295**

Precipitation in the cold, of a solution of cupric sulfate with potassium hydroxide gives the basic salt.

$$4CuSO_4 + 6KOH \rightarrow \underline{(CuO)_4SO_3} + (3K_2SO_4 + 3H_2O)$$

S. U. Pickering, Chem. News, **47**, 181 (1883)
Ref., C. F. M., Science, **2**, 19 (1883) *73*

$CuSO_4$

III-1296

KOH

$K_3C_6H_5O_7$

$H_3C_6H_5O_7$

A potassium cupricitrate in which the copper is electronegative is prepared by mixing potassium hydroxide, copper sulfate, potassium citrate, and citric acid. Copper acetate may be substituted for the copper sulfate.

(a) $3CuSO_4 + 2K_3C_6H_5O_7 + 18KOH + 4H_3C_6H_5O_7 \rightarrow 3[K_6Cu(C_6H_5O_7)_2 \cdot 6H_2O] + (3K_2SO_4 + 3H_2)$

(b) $3Cu(C_2H_3O_2)_2 + 2K_3C_6H_5O_7 + 18KOH + 4H_3C_6H_5O_7 \rightarrow 3[K_6Cu(C_6H_5O_7)_2 \cdot 6H_2O] + (6KC_2H_3O_2 + 3H_2)$

Luff, Z. Ges. Brauwesen, **21**, 319 (1898)
Ref., S. U. Pickering, J. Chem. Soc. (London), **97**, 1842 (1910) *67*

$CuSO_4$

K_2CO_3 **III-1297**

A solution of copper sulfate (1 g of Cu/l) is added to a small excess of potassium carbonate and boiled for a half hour precipitating copper oxide.

$$(CuSO_4 + K_2CO_3 \rightarrow K_2SO_4 + CO_2 + \underline{CuO})$$

W. Gibbs, Chem. News, **2**, 259 (1868) *101*

$CuSO_4$

K_2SO_4 **III-1298**

H_2O

Equimolecular quantities of cupric sulfate and potassium sulfate in water solution were mixed and allowed to crystallize in order to form potassium cupric sulfate hexahydrate.

$$K_2SO_4 + CuSO_4 + 6H_2O \rightarrow K_2Cu(SO_4)_2 \cdot 6H_2O$$

A. E. H. Tutton, Trans. Roy. Soc. (London), **216A**, 38 (1916) *105*

$CuSO_4$

$K_3Cr(CN)_6$ **III-1299**

A blue precipitate of cupric chromicyanide is formed when a solution of copper sulfate is treated with potassium chromicyanide.

$$3CuSO_4 + 2K_3Cr(CN)_6 \rightarrow \underline{Cu_3Cr_2(CN)_{12}} + (3K_2SO_4)$$

Kaiser, Jahresber., **17**, 302 (1864)
Ref., Cruser and Miller, J. Am. Chem. Soc., **28**, 1139 (1906) *1*

$CuSO_4$

$K_4Fe(CN)_6$ **III-1300**

The titration of copper by potassium ferrocyanide can be followed by the electrometric method under proper conditions.

$$2CuSO_4 + K_4Fe(CN)_6 \rightarrow \underline{Cu_2Fe(CN)_6} + 2K_2SO_4$$

P. Dutoit and G. Von Weisse, J. Chim. phys., **9**, 578 (1911) *69*

$CuSO_4$

$K_4Fe(CN)_6$ **III-1301**

Copper ferrocyanide is precipitated when copper sulfate, slightly acid with sulfuric acid, is treated with potassium ferrocyanide.

$$2CuSO_4 + K_4Fe(CN)_6 \rightarrow \underline{Cu_2Fe(CN)_6} + 2K_2SO_4$$

Matteo Spica, Staz. Sperim. Agric. ital., **26**, 593 (1894)
Ref., ibid., Chem. Centr., **65**, 815 (1894)
Ref., Fernekes and Koch, J. Am. Chem. Soc., **27**, 1231 (1905) *1*

$CuSO_4$

$K_4Fe(CN)_6$ **III-1302**

Copper sulfate and potassium ferrocyanide, both thoroughly dried, react when ground together.

$$2CuSO_4 + K_4Fe(CN)_6 \rightarrow Cu_2Fe(CN)_6 + 2K_2SO_4$$

L. H. Parker, J. Chem. Soc. (London), **105**, 1508 (1914) *57*

$CuSO_4$

Li_2SO_4 **III-1303**

No stable double salts exist in the system $CuSO_4$-$Li_2SO_4 \cdot H_2O$ at temperatures from 0–55°C. The solid phases are individual hydrates.

(a) $CuSO_4 + 5H_2O \rightarrow CuSO_4 \cdot 5H_2O$

(b) $Li_2SO_4 + H_2O \rightarrow Li_2SO_4 \cdot H_2O$

H. D. Crockford and M. M. Webster, J. Phys. Chem., **34**, 2377 (1930) *85*

$CuSO_4$

NH_2OH **III-1304**

Pale green microscopic crystals are obtained when methyl alcohol solutions of cupric sulfate and hydroxylamine are mixed.

$$CuSO_4 + NH_2OH \rightarrow \underline{CuSO_4 \cdot NH_2OH}$$

M. Adams and E. Overman, J. Am. Chem. Soc., **31**, 638 (1909) *1*

$CuSO_4$

NH_2OH **III-1305**

A deep green precipitate forms when methyl alcohol solution of cupric sulfate reacts with a slight excess of alcoholic hydroxylamine.

$$CuSO_4 + 2NH_2OH \rightarrow \underline{CuSO_4 \cdot 2NH_2OH}$$

M. Adams and E. Overman, J. Am. Chem. Soc., **31**, 638 (1909) *1*

$CuSO_4$

NH_3 **III-1306**

The following reactions embrace essentially the accepted ideas of the solubility of copper sulfate in excess ammonia.

(a) $4CuSO_4 \cdot 5H_2O + 6NH_4OH$
$\rightarrow CuSO_4 \cdot 3Cu(OH)_2 \cdot H_2O + 3(NH_4)_2SO_4 + 19H_2O$

(b) $CuSO_4 \cdot 3Cu(OH)_2 \cdot H_2O + 2NH_4OH$
$\rightarrow 4Cu(OH)_2 + (NH_4)_2SO_4 + H_2O$

(c) $Cu(OH)_2 + 2NH_4OH \rightarrow Cu(NH_3)_2(OH)_2 + 2H_2O$

(d) $Cu(NH_3)_2(OH)_2 + (NH_4)_2SO_4 \rightarrow Cu(NH_3)_4SO_4 \cdot H_2O + H_2O$

(e) $4Cu(NH_3)_4SO_4 \cdot H_2O + 5H_2O$
$\rightarrow CuSO_4 \cdot 3Cu(OH)_2 \cdot H_2O + 3(NH_4)_2SO_4 + 10NH_4OH$

E. B. Holland and G. M. Gilligan, J. Phys. Chem., **31**, 730 (1927) *85*

$CuSO_4$

NH_3 **III-1307**

When ammonia is added gradually to a copper sulfate solution, the following changes probably occur: (a) Copper hydroxide is precipitated along with ammonium sulfate. (b) Copper hydroxide dissolves in excess ammonia. (c) Complex base reacts further with ammonium sulfate.

(a) $CuSO_4 + 2NH_3 + 2H_2O \rightarrow \underline{(NH_4)_2SO_4 + Cu(OH)_2}$

(b) $Cu(OH)_2 + 2NH_3 \rightarrow Cu(OH)_2 \cdot 2NH_3$

(c) $Cu(OH)_2 \cdot 2NH_3 + (NH_4)_2SO_4 \rightarrow CuSO_4 \cdot 4NH_3 + 2H_2O$

H. M. Dawson and J. McCrae, J. Chem. Soc. (London), **77**, 1258 (1900) *102*

$CuSO_4$

NaOH **III-1308**

NH_3

Aqueous ammonia is added to boiling solution of cupric sulfate. Green basic cupric sulfate is precipitated. This when triturated with sodium hydroxide produces cupric hydroxide.

(a) $2CuSO_4 + 2NH_3 + 2H_2O \rightarrow \underline{Cu_2(OH)_2SO_4} + (NH_4)_2SO_4$

(b) $Cu_2(OH)_2SO_4 + 2NaOH \rightarrow 2Cu(OH)_2 + Na_2SO_4$

H. M. Dawson, J. Chem. Soc. (London), **95**, 374 (1909) *57*

$CuSO_4$

NH_4CNS **III-1309**

Pure white cuprous thiocyanate precipitates when copper sulfate, slightly acid with sulfuric acid, is saturated with carbon dioxide and then ammonium thiocyanate added.

$$2CuSO_4 + 2NH_4CNS \rightarrow \underline{2CuCNS} + (NH_4)_2SO_4 + SO_2 + (O_2)$$

Rivot, Z. anorg. Chem., **26**, 230 (1901)
Ref., Fernekes and Koch, J. Am. Chem. Soc., **27**, 1232 (1905) *1*

$CuSO_4$

III-1310

NH_4OH

$C_4O_5H_6$

The neutralization curve of mixtures of copper sulfate and malic acid by ammonia reveals the formation of a complex compound:

$$(CuSO_4 + C_4O_5H_6 + 6NH_4OH \rightarrow CuC_4O_5H_4(NH_3)_4 + (NH_4)_2SO_4 + 6H_2O)$$

J. L. Delsal, J. Chim. phys., **35**, 314 (1938) *69*

$CuSO_4$

III-1311

NH_4OH

$C_4O_6H_6$

Two complex compounds are formed in the neutralization of 1 mole of tartartic acid and 2 moles of copper sulfate by ammonia:

(a) $(2CuSO_4 + C_4O_6H_6 + 4NH_4OH \rightarrow CuC_4O_6H_4 + 2(NH_4)_2SO_4 + Cu(OH)_2 + 2H_2O)$

(b) $(2CuSO_4 + C_4O_6H_6 + 4NH_4OH \rightarrow Cu_2C_4O_6H_2 + 2(NH_4)_2SO_4 + 4H_2O)$

J. L. Delsal, J. Chim. phys., **35**, 314 (1938) *69*

$CuSO_4$

III-1312

NH_4OH

$C_4O_6H_6$

The titration curves of equimolecular solutions of copper sulfate and tartaric acid show the formation of the following complex compounds:

(a) $(CuSO_4 + C_4O_6H_6 + 2NH_4OH \rightarrow CuC_4O_6H_4 + (NH_4)_2SO_4 + 2H_2O)$

(b) $(CuSO_4 + C_4O_6H_6 + 3NH_4OH \rightarrow CuNH_4C_4O_6H_3 + (NH_4)_2SO_4 + 3H_2O)$

(c) $(CuSO_4 + C_4O_6H_6 + 4NH_4OH \rightarrow Cu(NH_4)_2C_4O_6H_2 + (NH_4)_2SO_4 + 4H_2O)$

(d) $(CuSO_4 + C_4O_6H_6 + 6NH_4OH \rightarrow CuC_4O_6H_4 \cdot 4(NH_3) + (NH_4)_2SO_4 + 6H_2O)$

J. L. Delsal, J. Chim. phys., **35**, 314 (1938) *69*

$CuSO_4$

$(NH_4)_2SO_4$ **III-1313**

When concentrated solutions of copper sulfate and ammonium sulfate are mixed, a blue solution is formed which when heated and diluted produces a bulky blue precipitate of basic sulfate.

$$15CuSO_4 + 14(NH_4)_2SO_4 + 14H_2O \rightarrow \underline{15CuO \cdot SO_3 \cdot 14NH_4HSO_4} + 14NH_4OH$$

S. V. Pickering, J. Chem. Soc. (London), **95:2**, 1417 (1909) *57*

$CuSO_4$

$(NH_4)_2SO_4$ **III-1314**

H_2O

Equimolecular quantities of cupric sulfate and ammonium sulfate in water solution were mixed and allowed to crystallize in order to form ammonium cupric sulfate hexahydrate.

$$(NH_4)_2SO_4 + CuSO_4 + 6H_2O \rightarrow (NH_4)_2Cu(SO_4)_2 \cdot 6H_2O$$

A. E. H. Tutton, Trans. Roy. Soc. (London), **216A**, 34 (1916) *105*

$CuSO_4$

NO **III-1315**

When copper sulfate is dissolved in concentrated sulfuric acid and nitric oxide is passed into the solution, a blue violet addition compound containing one mole nitric oxide per mole copper sulfate is formed. The addition compound is very sensitive toward water. Other anhydrous solvents may be used instead of concentrated sulfuric acid.

$$CuSO_4 + NO \rightarrow CuSO_4 \cdot NO$$

Manchot, Ann., **375**, 310 (1910) *41*

$CuSO_4$

$N_2H_4 \cdot 2H_2O$ **III-1316**

Copper sulfate may be reduced by hydrazine hydrate with the formation of a deposit of metallic copper.

(a) $CuSO_4 + N_2H_4 \cdot 2H_2O \rightarrow Cu(OH)_2 + N_2H_4 \cdot H_2SO_4$

(b) $2Cu(OH)_2 + N_2H_4 \cdot 2H_2O \rightarrow \underline{2Cu} + N_2 + 6H_2O$

Robert D. Barnard, Science, **66**, 330 (1927) *44*

$CuSO_4$

$NaCbO_3$ **III-1317**

A green copper salt precipitates when copper sulfate solution reacts with sodium columbate.

$$2CuSO_4 + 4NaCbO_3 + 7H_2O \rightarrow (2Na_2SO_4) + \underline{2(CuO \cdot Cb_2O_5 \cdot 3\tfrac{1}{2}H_2O)}$$

C. W. Balke and E. F. Smith, J. Am. Chem. Soc., **30**, 1652 (1908) *1*

$CuSO_4$

NaCl **III-1318**

ϵ

When a continuous current is passed through an aqueous series of electrolytes which does not contain a common ion, a chemical reaction occurs. In the series NaCl-$CuSO_4$-NaCl the copper sulfate solution is discolored at the left-hand contact while the color is intensified at the right-hand by the formation of cupric chloride.

$$CuSO_4 + 2NaCl + \epsilon \rightarrow CuCl_2 + Na_2SO_4$$

M. Chanoz, Compt. rend., **148**, 986–7 (1909) *38*

$CuSO_4$

NaCl **III-1319**

When a mixture of cupric sulfate and sodium chloride is heated, cupric chloride and sodium sulfate are formed.

$$CuSO_4 + 2NaCl \rightarrow CuCl_2 + Na_2SO_4$$

P. Klason, Sv. Kem. Tidsk., **29**, 192 (1917) *10*

$CuSO_4$

$NaHCO_3$ **III-1320**

When concentrated solutions of cupric sulfate and sodium bicarbonate are mixed a blue solution is formed, from which a double salt separates in 24 hours or more.

$$CuSO_4 + 2NaHCO_3 + 2H_2O \rightarrow \underline{CuSO_4 \cdot Na_2CO_3 \cdot 3H_2O} + CO_2$$

S. V. Pickering, J. Chem. Soc. (London), **95:2**, 1416 (1909) *57*

$CuSO_4$

$NaHSO_2 \cdot CH_2O$ **III-1321**

NH_4OH

A strongly ammoniacal copper sulfate solution reacts with a sodium formaldehyde-sulfate solution to give sodium ammonium sulfite, formaldehyde, cuprous sulfate, and ammonium sulfate.

$$NaHSO_2 \cdot CH_2O + 2CuSO_4 + 3NH_4OH \rightarrow Na(NH_4)SO_3 + CH_2O + Cu_2SO_4 + (NH_4)_2SO_4 + 2H_2O$$

Salkin, Ind. Eng. Chem., **15**, 849 (1923) *23*

$CuSO_4$

NaOH **III-1322**

$NaHSO_3$

Sodium bisulfite reacts with copper sulfate, in the presence of small amounts of sodium chloride and enough sodium hyroxide to neutralize sulfuric acid liberated, to give a precipitate of white crystalline cuprous chloride.

$$2CuSO_4 + NaHSO_3 + 2NaCl + 3NaOH \rightarrow 2CuCl + 3Na_2SO_4 + 2H_2O$$

L. P. Curtin, Ind. Eng. Chem., **19**, 993 (1927) *23*

$CuSO_4$

$NaHSO_3$ **III-1323**

Na_2CO_3

Sodium bisulfite reacts with copper sulfate in the presence of sodium chloride and sodium carbonate to yield sodium cuprous chloride.

$$4CuSO_4 + 2NaHSO_3 + 8NaCl + 3Na_2CO_3 \rightarrow 4Na(CuCl_2) + 6Na_2SO_4 + H_2O + 3CO_2$$

L. P. Curtin, Ind. Eng. Chem., **19**, 994 (1927) *23*

$CuSO_4$

$NaHSeO_3$ **III-1324**

When solutions of copper sulfate and of sodium hydrogen selenite are mixed, there precipitates a yellowish green amorphous salt which changes rapidly to blue, four-sided prisms of di-hydrated copper selenite.

$$CuSO_4 + NaHSeO_3 + 2H_2O \rightarrow \underline{CuSeO_3 \cdot 2(H_2O)} + (NaHSO_4)$$

L. F. Nilson, K. Sven. Vet. Ak. Handl. Övers., **31**, No. 1, 42 (1874) *10*

$CuSO_4$

NaI **III-1325**

Sodium iodide reduces sulfate to cuprous iodide, setting some iodine free and leaving sodium sulfate in solution.

$$2CuSO_4 + 4NaI \rightarrow 2Na_2SO_4 + \underline{2CuI} + I_2$$

G. Holst, Sv. Kem. Tidsk., **43**, 16 (1931) *10*

$CuSO_4$

$C_6H_4(OH)_2$ **III-1326**

Light

Red light from a carbon arc causes reduction of Fehling's solution containing about a quarter of a gram of hydroquinone per liter, whereas Fehling's solution without hydroquinone is not affected during the same interval of illumination.

$$2CuSO_4 + C_6H_4(OH)_2 + H_2O \rightarrow \underline{Cu_2O} + (C_6H_4O_2 + 2H_2SO_4)$$

A. Leighton, J. Phys. Chem., **17**, 203 (1913) *7*

$CuSO_4$

NaOH **III-1327**

Addition of .1*N* sodium hydroxide solution to .1*N* cupric sulfate solution results in a precipitate of basic cupric sulfate.

$$4CuSO_4 + 6NaOH \rightarrow \underline{(Cu(OH)_2)_3 \cdot CuSO_4} + 3Na_2SO_4$$

R. H. Fuoss, Anal. Ed., **1**, 128 (1929) *33*

$CuSO_4$

NaOH **III-1328**

ε

When a continuous current is passed through an aqueous series of electrolytes, which does not contain a common ion, a chemical reaction occurs. In the series NaOH-$CuSO_4$-NaOH the blue precipitate of cupric hydroxide increases at the right-hand contact.

$$CuSO_4 + 2NaOH \rightarrow \underline{Cu(OH)_2} + Na_2SO_4$$

M. Chanoz, Compt. rend., **148**, 987 (1909) *88*

$CuSO_4$

NaOH **III-1329**

The composition of basic cupric sulfates is dependent on the methods of preparation and the ratio between the reactants. The composition remains essentially $7CuO \cdot 2SO_3$ until a pH of about 7, and thereafter a continuous increase of CuO/SO_3 results.

$$7CuSO_4 + 10NaOH \rightarrow (CuO)_7 \cdot 2SO_3 + 5Na_2SO_4 + 5H_2O$$

O. A. Nelson, J. Phys. Chem., **32**, 1189 (1928) *86*

$CuSO_4$

NaOH **III-1330**

The precipitate obtained by adding not more than 1.5 moles of NaOH to each mole of $CuSO_4$ is practically constant in composition.

$$4CuSO_4 + 6NaOH + H_2O \rightarrow CuSO_4\,3CuO \cdot 4H_2O + 3Na_2SO_4$$

F. S. Williamson, J. Phys. Chem., **27**, 796 (1923) *86*

$CuSO_4$

NaOH **III-1331**

$C_4O_5H_6$

The electrometric titration curve of an equimolecular solution of copper sulfate and malic acid reveals the existence of a complex compound:

$$(CuSO_4 + C_4O_5H_6 + 3NaOH \rightarrow CuNaC_4O_5H_3 + Na_2SO_4 + 3H_2O)$$

J. L. Delsal, J. Chim. phys., **35**, 314 (1938) *69*

$CuSO_4$

III-1332

NaOH

$C_4O_5H_6$

A crystalline addition compound can be obtained when a solution of 2 moles of malic acid and 3 moles of copper sulfate is neutralized by sodium hydroxide:

$$(3CuSO_4 + 2C_4O_5H_6 + 6NaOH \rightarrow Cu_3(C_4O_5H_3)_2 + 3Na_2SO_4 + 6H_2O)$$

J. L. Delsal, J. Chim. phys., **35**, 314 (1938) *69*

$CuSO_4$

III-1333

NaOH

$KNaC_4O_6H_4$

Two complex compounds are formed in the neutralization of equimolecular solutions of copper sulfate and Rochelle salt by sodium hydroxide:

(a) $(CuSO_4 + KNaC_4O_6H_4 + NaOH \rightarrow KCuC_4O_6H_3 + Na_2SO_4 + H_2O)$

(b) $(CuSO_4 + KNaC_4O_6H_4 + 2NaOH \rightarrow KNaCuC_4O_6H_2 + Na_2SO_4 + 2H_2O)$

J. L. Delsal, J. Chim. phys., **35**, 314 (1938) *69*

$CuSO_4$

III-1334

NaOH

$Na_2C_4O_6H_4$

Two complex compounds are formed in the neutralization of a solution of 1 mole of sodium tartrate and 2 moles of copper sulfate with sodium hydroxide:

(a) $(2CuSO_4 + Na_2C_4O_6H_4 + 3NaOH \rightarrow Cu_2ONaC_4O_6H_3 + 2Na_2SO_4 + 2H_2O)$

(b) $(Cu_2ONaC_4O_6H_3 + NaOH \rightarrow CuNa_2C_4O_6H_2 + \underline{Cu(OH)_2})$

J. L. Delsal, J. Chim. phys., **35**, 314 (1938) *69*

$CuSO_4$

NaOH **III-1335**

Na_2CO_3

On mixing copper sulfate with sodium carbonate solution (1 mole of sulfate for .93 mole of carbonate) the sulfate is completely converted into three distinct compounds: Insoluble hydrated copper carbonate which forms the bulk of the precipitate, insoluble hydrated basic copper sulfate, and soluble basic copper sulfate. These compounds are formed in the so-called "Burgundy mixture" and the percentage composition of the precipitate varies greatly with temperature, etc. If sodium bicarbonate is used, the same compounds are formed.

$$(13CuSO_4 \cdot 5H_2O + 3Na_2CO_3 \cdot 10H_2O + 14NaOH \rightarrow \underline{3CuCO_3 \cdot 3Cu(OH)_2 \cdot H_2O} + \underline{Cu_5(SO_4)_2(OH)_6 \cdot 2H_2O} + CuSO_4 \cdot Cu(OH)_2 + 10Na_2SO_4 + 92H_2O)$$

Robert L. Mond and Christian Heberlein, J. Chem. Soc. (London), **115**:2, 921 (1919) *48*

$CuSO_4$

Na_2CO_3 **III-1336**

Burgundy mixture is made by adding a cool, dilute solution of copper sulfate to an equivalent amount of cool, dilute sodium carbonate. The precipitate is then washed free of soluble salt.

$$5CuSO_4 \cdot 5H_2O + 8Na_2CO_3 \cdot 10H_2O \rightarrow 5CuO \cdot 2CO_2 \cdot 6H_2O + 5Na_2SO_4 \cdot 10H_2O + 6NaHCO_3 + 46H_2O$$

E. B. Holland and G. M. Gilligan, J. Phys. Chem., **31**, 731 (1927) *85*

$CuSO_4$

Na_2CO_3 **III-1337**

When copper sulfate and sodium carbonate are mixed, a bulky blue precipitate is thrown down. When the precipitate remains in contact with a saturated solution of sodium carbonate several days, it changes to cupric hydroxide.

(a) $5CuSO_4 + 8Na_2CO_3 + 3H_2O \rightarrow \underline{5CuO \cdot 2CO_2} + 5Na_2SO_4 + 6NaHCO_3$

(b) $5CuO \cdot 2CO_2 + 2Na_2CO_3 + 7H_2O \rightarrow \underline{5Cu(OH)_2} + (4NaHCO_3)$

S. V. Pickering, J. Chem. Soc. (London), **95**:2, 1412 (1909) *57*

$CuSO_4$

III-1338

Na_2CO_3

In the preparation of mixed Cu-Ni catalyst for oil hydrogenation, the copper is precipitated, (with the Ni), from a solution containing 0.5–0.8% of the mixed sulfates, by adding dilute Na_2CO_3 soln, the temp. being held at 40°C. After adding kieselghur as an adsorbent, the mixed carbonates are filtered and dried.

$$CuSO_4 + Na_2CO_3 \rightarrow \underline{CuCO_3} + Na_2SO_4$$

Masloboino Zhirovoe Delo, **13**, No. 4, 31–34 (1937)
Ref., Abstracts, in Oil & Soap, **15:3**, 77 (1938) *111*

$CuSO_4$

III-1339

Na_2CO_3

In a method of recovery of metallic mixtures used as catalysts, (Ni-Cu mixtures), the metals are dissolved in nine percent sulfuric acid, then precipitated with sodium carbonate.

(a) $2Cu + 2H_2SO_4 + O_2(\text{from air}) \rightarrow 2CuSO_4 + 2H_2O$

(b) $2CuSO_4 + 2Na_2CO_3 + H_2O \rightarrow Cu_2(OH)_2CO_3 + 2Na_2SO_4 + CO_2$

V. Kopeikovskii and T. Lapina, Masloboino Zhirovoe Delo, **12**, 580–581
Ref., Rev. of Sci. Lit. on Fats & Oils, Oil and Soap, **15:4**, 95 (1938) *111*

$CuSO_4$

III-1340

Na_2CO_3

A saturated solution of pure cupric sulfate is diluted with an equal quantity of water, and treated with a solution containing 5% sodium carbonate and 5% sodium bicarbonate. The mixture is allowed to stand and then filtered. A basic copper carbonate is formed.

$$7CuSO_4 + 12Na_2CO_3 + 10H_2O \rightarrow 2CuCO_3 \cdot 5Cu(OH)_2 + 10NaHCO_3 + 7Na_2SO_4$$

H. B. Dunnicliff and S. Lal, J. Chem. Soc. (London), **113**, 718 (1918) *57*

$CuSO_4$

$Na_2C_2O_4$ **III-1341**

When a solution of copper sulfate is treated with an equivalent amount of sodium oxalate, copper oxalate precipitates. The copper oxalate is blue and is hydrated. The amount of hydrate water varies with conditions. The most stable hydrate is $CuC_2O_4 \cdot \frac{1}{2}H_2O$.

$$CuSO_4 + Na_2C_2O_4 \rightarrow \underline{CuC_2O_4} + (Na_2SO_4)$$

K. Seubert and G. Rauter, Ber., **25**, 2821 (1892) *91*

$CuSO_4$

Na_2O_2 **III-1342**

Copper peroxide is unstable in alkaline solution. When copper sulfate is oxidized with sodium peroxide, the resulting copper peroxide will decompose yielding copper oxide.

(a) $$CuSO_4 + Na_2O_2 \rightarrow CuO_2 + Na_2SO_4$$

(b) $$2CuO_2 + 2H_2O \rightarrow 2Cu(OH)_2 + O_2$$

(c) $$Cu(OH)_2 \rightarrow CuO + H_2O$$

L. Moser, Z. anorg. Chem., **54**, 121 (1907) *28*

$CuSO_4$

$Na_2S_2O_3$ **III-1343**

NH_4OH

When a concentrated solution of sodium thiosulfate is added to an ammoniacal solution of a copper salt and the mixture allowed to stand for a short time, a mass of deep blue crystals of cuprous sodium-ammonium thiosulfate separate.

$$8Na_2S_2O_3 + 6CuSO_4 + 12NH_4OH$$
$$\rightarrow \underline{(Cu_2S_2O_3)_3 \cdot (Na_2S_2O_3)_4 \cdot (NH_4)_2S_2O_3 \cdot 6NH_3} + (2(NH_4)_2SO_4 + 9H_2O + 4Na_2SO_4 + 3O)$$

O. L. Shinn, J. Am. Chem. Soc., **26**, 948 (1904) *1*

$CuSO_4$

$Na_2S_2O_3$ **III-1344**

When sodium thiosulfate is added to a cupric salt, Cu_2S and CuS are formed in varying quantities depending upon temperature, duration of heating, amount of $Na_2S_2O_3$ added, and acidity. On boiling a slightly acid solution with an excess of $Na_2S_2O_3$, the following equation is practically complete:

$$(2CuSO_4 + 2Na_2S_2O_3 + 2H_2O \rightarrow \underline{Cu_2S} + 2Na_2SO_4 + 2H_2SO_4 + S)$$

J. Hanus and V. Horvorka, Coll. Czech. Comm., **1**, 65 (1929) *87*

$CuSO_4$

$Na_2S_2O_3$ **III-1345**

In the titration of a cupric salt with alkaline thiosulfate, the first step is the reduction to the cuprous state.

$$2CuSO_4 + 2Na_2S_2O_3 \rightarrow Cu_2S_4O_6 + 2Na_2SO_4$$

Bhaduri, Z. anorg. Chem., **17**, 1 (1898)
Ref., P. Dutoit, J. Chim. phys., **11**, 650 (1913) *69*

$CuSO_4$

$Na_2S_2O_3$ **III-1346**

A solution of cupric sulfate will react with sodium thiosulfate; crystals of copper sodium thiosulfate are obtained. The same type reaction occurs with potassium thiosulfate and ammonium thiosulfate.

(a) $$20CuSO_4 + 18Na_2S_2O_3 + 17H_2O \rightarrow \underline{10Cu_2S_2O_3 \cdot 8Na_2S_2O_3 \cdot 17H_2O} + 10Na_2S_2O_8$$

(b) $$2CuSO_4 + 2K_2S_2O_3 + 2H_2O \rightarrow \underline{Cu_2S_2O_3 \cdot K_2S_2O_3 \cdot 2H_2O} + K_2S_2O_8$$

(c) $$4CuSO_4 + 6(NH_4)_2S_2O_3 + 3H_2O \rightarrow \underline{2Cu_2S_2O_3 \cdot 4(NH_4)_2S_2O_3 \cdot 3H_2O} + 2(NH_4)_2S_2O_8$$

A. Rosenheim and S. Steinhauser, Z. anorg. Chem., **25**, 72 (1900) *28*

$CuSO_4$

$Na_2S_2O_4$ **III-1347**

An aqueous solution of copper sulfate added to an excess of a solution of sodium dithionite changes color and a red brown precipitate is formed together with the evolution of a gas. The precipitate is a mixture of copper and cupric sulfide.

(a) $CuSO_4 + Na_2S_2O_4 \rightarrow Cu + Na_2SO_4 + 2SO_2$

(b) $Cu + 2Na_2S_2O_4 \rightarrow CuS + Na_2S_2O_5 + Na_2SO_3$

O. Brunck, Ann., **327**, 243 (1903) *20*

$CuSO_4$

$Na_2S_2O_4$ **III-1348**

NH_4OH

A strongly ammoniacal copper sulfate solution reacts with a sodium hyposulfite solution to give sodium sulfite, cuprous sulfate, ammonium sulfite and ammonium sulfate.

$$Na_2S_2O_4 + 2CuSO_4 + 4NH_4OH \rightarrow Na_2SO_3 + Cu_2SO_4 + (NH_4)_2SO_3 + (NH_4)_2SO_4 + 2H_2O$$

B. Salkin, Ind. Eng. Chem., **15**, 849 (1923) *23*

$CuSO_4$

Na_2SiO_3 **III-1349**

H_2O

From M/10 cupric sulfate and M/10 Na_2SiO_3 solutions at the boiling point, light bluish green to deep blue precipitates, are thrown down. When the molecular proportion of $CuSO_4$ to Na_2SiO_3 is not less than 1 to 1, x-ray analysis shows the product to be a basic copper sulfate $4CuO \cdot SO_3 \cdot 3H_2O$ (a). Sometimes the product is (a) mixed with $3CuO \cdot SO_3 \cdot 2H_2O$. All the precipitates occlude silica.

$$4CuSO_4 + 2Na_2SiO_3 + 6H_2O \rightarrow \underline{4CuO \cdot SO_3 \cdot 3H_2O} + (2Na_2SO_4 + H_2SO_4 + 2H_2SiO_3)$$

H. V. Anderson and F. P. Hochgesang, J. Phys. Chem., **44**, 439 (1940) *7*

$CuSO_4$

III-1350

$Na_4P_2O_7$

When solutions of copper sulfate and sodium pyrophosphate are mixed, there precipitates a bluish white with a tinge of green, amorphous, probably anhydrous, pyrophosphate of copper. Two different hydrated pyrophosphates of copper are known.

(a) $2CuSO_4 + Na_4P_2O_7 \rightarrow \underline{Cu_2P_2O_7} + (2Na_2SO_4)$

(b) $2Cu_2P_2O_7 + 5H_2O \rightarrow (Cu_2P_2O_7)_2 \cdot (H_2O)_5$

(c) $Cu_2P_2O_7 + 5H_2O \rightarrow Cu_2P_2O_7 \cdot (H_2O)_5$

C. N. Pahl, K. Sven. Vet. Ak. Handl. Övers., **30**, No. 7, 40 (1873) *10*

$CuSO_4$

III-1351

$Na_4P_2O_7$

H_2S

Copper pyrophosphate precipitates when pure cupric sulfate is added to a solution of sodium pyrophosphate, and is converted into pyrophosphoric acid by hydrogen sulfide.

(a) $2CuSO_4 + Na_4P_2O_7 \rightarrow \underline{Cu_2P_2O_7} + (2Na_2SO_4)$

(b) $Cu_2P_2O_7 + 2H_2S \rightarrow H_4P_2O_7 + \underline{2CuS}$

G. A. Abbott and W. C. Bray, J. Am. Chem. Soc., **31**, 734 (1909) *1*

$CuSO_4$

III-1352

$Na_5P_3O_{10}$

A solution of copper sulfate will react with a solution of sodium triphosphate yielding green crystals.

(a) $CuSO_4 + Na_5P_3O_{10} \rightarrow Na_3CuP_3O_{10} + Na_2SO_4$

(b) $2Na_3CuP_3O_{10} + CuSO_4$
$\rightarrow \underline{Cu_2P_2O_7 \cdot CuNa_2P_2O_7} + 2NaPO_3 + Na_2SO_4$

M. Stange, Z. anorg. Chem., **12**, 444 (1896) *28*

$CuSO_4$

$Na_8Fe_2(P_2O_7)_3$ **III-1353**

When a neutral or slightly acid copper sulfate solution is added to a solution of sodium ferropyrophosphate, cuprous oxide is formed in colloidal suspension and the acid solution contains sodium ferripyrophosphate.

$$2CuSO_4 + Na_8Fe_2(P_2O_7)_3 + H_2O \rightarrow \underline{Cu_2O} + 2NaHSO_4 + Na_6Fe_2(P_2O_7)_3$$

P. Pascal, Compt. rend., **146**, 863 (1908) *38*

$CuSO_4$

$Na_8Fe_2(P_2O_7)_3$ **III-1354**

NaI

Sodium ferropyrophosphate and sodium iodide (or bromide) precipitate copper from its solutions as cuprous iodide (or bromide).

$$2CuSO_4 + 2NaI + Na_8Fe_2(P_2O_7)_3 \rightarrow \underline{2CuI} + (2Na_2SO_4 + Na_6Fe_2(P_2O_7)_3)$$

P. Pascal, Compt. rend., **146**, 863 (1908) *38*

$CuSO_4$

P **III-1355**

Copper is precipitated when phosphorus reacts with a solution of cupric sulfate.

$$5CuSO_4 + 2P + 8H_2O \rightarrow 5Cu + 5H_2SO_4 + 2H_3PO_4$$

R. M. Bird and S. H. Diggs, J. Am. Chem. Soc., **36**, 1386 (1914) *1*

$CuSO_4$

P **III-1356**

Light

A copper sulfate solution to which a little phosphorus in ether has been added is more quickly reduced to metal and phosphide in the presence of light than in darkness:

$$3CuSO_4 + P \rightarrow \underline{Cu_3P} + \text{(other products)}$$

C. W. Bennett, J. Phys. Chem., **16**, 782 (1912) *7*

$CuSO_4$

III-1357

PH_3

When phosphine is passed into an aqueous solution of copper sulfate, copper phosphide and copper are precipitated.

(a) $8CuSO_4 + PH_3 + 4H_2O \rightarrow H_3PO_4 + 4H_2SO_4 + 4Cu_2SO_4$

(b) $3Cu_2SO_4 + 2PH_3 \rightarrow 3H_2SO_4 + 2Cu_3P$

(c) $4Cu_2SO_4 + PH_3 + 4H_2O \rightarrow H_3PO_4 + 4H_2SO_4 + 8Cu$

P. Kulisch, Ann., **231**, 327 (1885) *20*

$CuSO_4$

III-1358

P_4

H_2O

Phosphorus reacts with a concentrated solution of cupric sulfate in the absence of air.

$$3CuSO_4 + P_4 + 6H_2O \rightarrow Cu_3P_2 + 2H_3PO_3 + 3H_2SO_4$$

Schiff, Ann., **114**, 200 (1860) *26*

$CuSO_4$

III-1359

$Pb(CH_3SO_4)_2$

Solutions of copper sulfate and lead methyl sulfate are mixed, the lead sulfate filtered off, and the filtrate yields large, thin, blue, right angled tablets of tetra-hydrated copper (cupric) methyl sulfate.

$$CuSO_4 + Pb(CH_3SO_4)_2 + 4H_2O \rightarrow \underline{PbSO_4} + Cu(CH_3SO_4)_2 \cdot 4(H_2O)$$

J. E. Alèn, K. Sven. Vet. Ak. Handl. Övers, **37**, No. 8, 15 (1880) *10*

$CuSO_4$

III-1360

SO_2

NH_4OH

If sulfur dioxide is led into ammoniacal copper sulfate solution, the colorless, anhydrous double sulfite crystallizes out.

$$2CuSO_4 + 8NH_4OH + 3SO_2 \rightarrow \underline{(NH_4)_2Cu_2(SO_3)_2} + 3(NH_4)_2SO_4 + 4H_2O$$

Ramberg, Z. physik. Chem., **69**, 512 (1909)
J. L. Howe, J. Am. Chem. Soc., **33**, 168 (1911) *1*

$CuSO_4$

$SnCl_2$ **III-1361**

Stannous chloride reduces cupric sulfate and yields olive-green copper suboxide.

$$8CuSO_4 + 6SnCl_2 \rightarrow \underline{2Cu_4O} + (3SnCl_4 + 3SnO_2 + 8SO_3)$$

Å. G. Ekstrand, Sv. Kem. Tidsk., **10**, 29 (1898) *10*

$CuSO_4$

Zn **III-1362**

In order to prepare pure copper, zinc was added to cupric sulfate solution.

$$CuSO_4 + Zn \rightarrow \underline{Cu} + (ZnSO_4)$$

T. J. Baker, Trans. Roy. Soc. (London), **196**, 533 (1901) *105*

$CuSO_4$

ε **III-1363**

The powder and spongy-like cathodal deposits of copper formed by electrolysis of aqueous solutions at high current densities (.02 amp. per sq. cm.) are due to admixed oxides, formed by secondary processes at the cathode.

(a) $$2CuSO_4 + 2H_2O \rightarrow 2Cu + 2H_2SO_4 + O_2$$

(b) $$CuSO_4 + H_2O \rightarrow CuO + H_2SO_4$$

O. K. Kudra and K. N. Ivanov, J. Phys. Chem., **40**, 692 (1936) *85*

$CuSO_4$

ε **III-1364**

In order to prepare pure copper, a solution of copper sulfate was electrolyzed.

$$2CuSO_4 + 2H_2O \rightarrow 2Cu + (2H_2SO_4 + O_2)$$

W. A. Tilden, Trans. Roy. Soc. (London), **194**, 242 (1900) *105*

$CuSO_4$

III-1365

ε

Cupric sulfate was electrolyzed with copper electrodes. Copper and oxygen were formed.

(a) $CuSO_4 \rightarrow Cu + SO_4$

(b) $2SO_4 + 2H_2O \rightarrow 2H_2SO_4 + O_2$

J. W. Mallet, Trans. Roy. Soc. (London), **180**, 419 (1889) *105*

$CuSO_4$

III-1366

Δ

Above 600°C cupric sulfate is decomposed into the oxide:

$$(CuSO_4 \rightarrow CuO + SO_3)$$

H. O. Hoffman and W. Wanjukow, Trans. Am. Inst. Min. Eng., **43**, 523 (1913)
Ref., G. Marchal, J. Chim. phys., **22**, 325 (1925) *69*

$CuSO_4$

Ammonium heptylacetate **III-1367**

A solution of ammonium-heptylacetate is warmed and an aqueous solution of copper sulfate added as long as a precipitate forms. The precipitate is dissolved in hot alcohol and water added until a slight turbidity persists. As the solution cools crystals of copper heptylacetate form.

A similar reaction occurs when this reagent is treated in a similar fashion with zinc sulfate.

$$2C_8H_{17}COONH_4 + CuSO_4 \rightarrow \underline{Cu(C_8H_{17}COO)_2} + (NH_4)_2SO_4$$

F. Jourdan, Ann., **200**, 110–1 (1880) *20*

$CuSO_4$

Barium β-bromo isocymol sulfonate **III-1368**

Barium betabromo isocymol sulfonate is dissolved in water and the calculated quantity of copper sulfate dissolved in water is added. The solution is filtered and cooled. Crystals of copper β-bromo isocymol sulfonate with four H_2O separate.

$$Ba(C_{10}H_{12}BrSO_3)_2 + CuSO_4 \rightarrow Cu(C_{10}H_{12}BrSO_3)_2 + BaSO_4$$

W. Kelbe and N. Czarnomski, Ann., **235**, 275 (1886) *20*

$CuSO_4$

Calcium-3-bromo-2-toluene sulfonate **III-1369**

An aqueous solution of the calcium salt of 3-bromo-2-toluene sulfonic acid is treated with an aqueous solution of copper sulfate as long as a precipitate forms. The insoluble calcium sulfate was filtered off and the filtrate concentrated. Crystals of copper-3-bromo-2-toluenesulfonate with four H_2O separate.

$$Ca(C_7H_6BrSO_3)_2 + CuSO_4 \rightarrow \underline{Cu(C_7H_6BrSO_3)_2} + CaSO_4$$

E. Grete, Ann., **177**, 241 (1875) *20*

$CuSO_4$

Lead glycerate **III-1370**

A boiling solution of lead glycerate is treated with an aqueous solution of the equivalent amount of copper sulfate, the solution filtered and concentrated. An amorphous precipitate of copper glycerate precipitates.

A similar reaction occurs when lead glycerate is treated with a solution of $MnSO_4$. The manganous salt crystallizes with one half H_2O.

$$(C_2(OH)_2H_3COO)_2Pb + CuSO_4 \rightarrow \underline{Cu(C_3H_5O_4)_2} + PbSO_4$$

K. Garzarolli, Ann., **182**, 190 (1876) *20*

$CuSO_4$

Mono-lead-3-chloro-4-phenolsulfonate **III-1371**

An alcoholic solution of mono-lead-3-chloro-4-phenolsulfonate is treated with an aqueous solution of copper sulfate as long as a precipitate forms. The precipitated copper salt is recrystallized from water and contains four H_2O.

$$Pb(C_6H_4ClSO_4)_2 + CuSO_4 \rightarrow \underline{Cu(C_6H_4ClSO_4)_2} + PbSO_4$$

J. Kramers, Ann., **173**, 339 (1874) *20*

$CuSO_4$

Potassium anisate **III-1372**

Copper anisate may be prepared by adding a solution of copper sulfate to a solution of potassium anisate, obtained from anisic acid with potassium hydroxide. The copper salts of margaric, cinnamic, phthalic, abietic and camphoric acids have been made in a similar manner.

(a) $C_7H_7OCOOH + KOH \rightarrow C_7H_7OCOOK + H_2O$

(b) $2C_7H_7OCOOK + CuSO_4 \rightarrow \underline{(C_7H_7OCOO)_2Cu} + K_2SO_4$

C. B. Gates, J. Phys. Chem., **15**, 97 (1911) *7*

$CuSO_4$

Sodium benzoate **III-1373**

Copper benzoate was prepared by the reaction of copper sulfate upon sodium benzoate. It precipitates from water as a blue powder, insoluble in alcohol, slightly soluble in acetone, soluble in dioxane. Copper salicylate (b) was similarly prepared.

(a) $CuSO_4 + 2NaC_6H_5COO + 2H_2O \rightarrow Na_2SO_4 + \underline{(C_6H_5COO)_2Cu \cdot 2H_2O}$

(b) $CuSO_4 + 2C_6H_4OHCOONa \cdot 2H_2O \rightarrow Na_2SO_4 + \underline{(C_6H_4OHCOO)_2Cu \cdot 2H_2O}$

Berry, Hartung and Grubb, J. Am. Pharm. Assn., **31**, 43 (1942) *112*

$CuSO_4$

Sodium phenylmandelate **III-1374**

An aqueous solution of sodium phenylmandelate is treated with an aqueous solution of cupric sulfate and the residue filtered off and dried. A similar reaction occurs when this reagent is treated with silver nitrate.

(a) $2C_{13}H_{11}OCOONa + CuSO_4 \rightarrow (C_{13}H_{11}OCOO)_2Cu + Na_2SO_4$

(b) $C_{13}H_{11}OCOONa + AgNO_3 \rightarrow (C_{13}H_{11}OCOOAg) + NaNO_3$

R. Meyer, Ann., **220**, 52–3 (1883) *20*

$CuSO_4 \cdot 5H_2O$

I_2

SO_2 **III-1375**

CH_3OH

With cupric sulfate pentahydrate, a methanol solution of Karl Fischer reagent reduces the copper to the cuprous form.

$$CuSO_4 \cdot 5H_2O + 4\tfrac{1}{2}I_2 + 5SO_2 + 5CH_3OH \rightarrow CuI + 8HI + 5HSO_4CH_3 + H_2SO_4$$

J. Mitchell, Jr., D. M. Smith, E. C. Ashby and W. M. D. Bryant, J. Am. Chem. Soc., **63**, 2929 (1941) *16*

$CuSO_4 \cdot 5H_2O$

$(NH_2OH)_2 \cdot 2HCl$

KOH **III-1376**

Cuprous oxide is produced when a solution of copper sulfate is treated with a solution containing a mixture of hydroxylamine hydrochloride and potassium hydroxide.

$$(NH_2OH)_2 \cdot 2HCl + 4(CuSO_4 \cdot 5H_2O) + 10KOH \rightarrow \underline{2Cu_2O} + N_2O + 2KCl + 4K_2SO_4 + 29H_2O$$

Griggi, Boll. chim. farm., **43**, 392
Ref., J. Am. Chem. Soc., **27**, 1354 (1905) *1*

$CuSO_4 \cdot 5H_2O$

$(NH_4)_2SO_4$ **III-1377**

The soluble salt, copper ammonium sulfate, is prepared by melting one part of copper sulfate with three parts of ammonium sulfate and driving off the excess of the latter by gentle heat.

$$2CuSO_4 \cdot 5H_2O + (NH_4)_2SO_4 \rightarrow (CuSO_4)_2 \cdot (NH_4)_2SO_4 + 10H_2O$$

F. R. Mallet, J. Chem. Soc. (London), **77**, 216 (1900) *102*

$CuSO_4 \cdot 2NH_2OH$

NH_2OH **III-1378**

A double salt is produced when a concentrated solution of hydroxylamine is added to $CuSO_4 \cdot 2NH_2OH$.

$$CuSO_4 \cdot 2NH_2OH + 3NH_2OH \rightarrow \underline{CuSO_4 \cdot 5NH_2OH}$$

M. Adams and E. Overman, J. Am. Chem. Soc., **31**, 640 (1909) *1*

$CuSO_4 \cdot 5NH_2OH$

H_2O **III-1379**

A double salt is produced when water is added to $CuSO_4 \cdot 5NH_2OH$.

$$2(CuSO_4 \cdot 5NH_2OH) + H_2O \rightarrow \underline{Cu_2OSO_4 \cdot 2NH_2OH} + 6NH_2OH + (NH_2OH)_2H_2SO_4$$

M. Adams and E. Overman, J. Am. Chem. Soc., **31**, 640 (1909) *1*

$CuSO_4 \cdot 2NH_3$

H_2O_2 **III-1380**

When hydrogen peroxide is added to copper sulfate diammoniate, oxygen is evolved and an olive green precipitate of hydrated copper dioxide is formed.

$$2(CuSO_4 \cdot 2NH_3) + 4H_2O_2 \rightarrow 2CuO_2 \cdot H_2O + O_2 + 2(NH_4)_2SO_4$$

C. Weltzien, Compt. rend., **63**, 519 (1866) *29*

$(CuSO_4 \cdot 4NH_3)$

KCN **III-1381**

Copper in ammonical solution can be quantitatively determined by titrating to the disappearance of the blue color.

(a) $CuSO_4(NH_3)_4 + 2KCN \rightarrow Cu(CN)_2 + K_2SO_4 + 4NH_3$

(b) $2Cu(CN)_2 \rightarrow 2CuCN + (CN)_2$

V. Lafollye,
Ref., A. Henninger, Ber., **5**, 431 (1872) *11*

$CuSO_4 \cdot 4NH_3$

KCN **III-1382**

A potassium copper cyanide forms when ammoniacal copper sulfate is treated with potassium cyanide.

$$2[CuSO_4 \cdot 4NH_3] + 7KCN + 7H_2O \rightarrow 2K_2Cu(CN)_3 + KCNO + K_2SO_4 + (NH_4)_2SO_4 + 6NH_4OH$$

O. L. Barnebey, J. Am. Chem. Soc., **36**, 1092 (1914) *1*

$CuSO_4 \cdot 4NH_3$

$(N_2H_4)H_2SO_4$ **III-1383**

Hydrazine sulfate reduces cupric sulfate tetrammoniate to the corresponding cuprous salt.

$$4CuSO_4(NH_3)_4 + (N_2H_4 \cdot H_2SO_4) \rightarrow 2Cu_2SO_4(NH_3)_4 + N_2 + 2NH_3 + 3(NH_4)_2SO_4$$

Ebler, Z. anorg. Chem., **47**, 371 (1906)
Ref., J. Am. Chem. Soc., **28**, 1763 (1906) *1*

$CuSO_4 \cdot 4NH_3 \cdot H_2O$

KI **III-1384**

When aquocopper sulfate tetrammoniate is treated with potassium iodide in iodine solution; tetraquo cuprous iodide monoammoniate, potassium sulfate, iodine and ammonia are produced.

$$2CuSO_4 \cdot 4NH_3 \cdot H_2O + 4KI + 2H_2O \rightarrow 2K_2SO_4 + Cu_2I_2 \cdot NH_3 \cdot 4H_2O + I_2 + 7NH_3$$

O. Silberrad, J. Chem. Soc. (London), **87**, 67 (1905) *102*

$CuSO_4 \cdot 5NH_3$

H_2O **III-1385**

When damp air is passed over copper sulfate pentammoniate the ammonia is replaced by water.

$$CuSO_4 \cdot 5NH_3 + 5H_2O \rightarrow CuSO_4 \cdot 5H_2O + 5NH_3$$

D. Mendelejeff,
Ref., Von Richter, Ber., **3**, 422 (1870) *11*

$CuS_2O_6 \cdot 4NH_3$

NH_3 **III-1386**

Copper dithionate tetrammoniate is reacted upon with gaseous ammonia at low temperature. Copper dithionate hexammoniate is formed.

$$CuS_2O_6 \cdot 4NH_3 + 2NH_3 \rightarrow \underline{Cu(S_2O_6) \cdot 6NH_3}$$

Fritz Ephraim and Edward Bolle, Ber., **48**, 1770 (1916) *28*

CuS_4O_6

NH_3 **III-1387**

Crystals of bright blue color are obtained when a concentrated solution of copper tetrathionate is saturated with ammonia gas.

$$CuS_4O_6 + 4NH_3 \rightarrow \underline{CuS_4O_6 \cdot 4NH_3}$$

Fritz Ephraim and Edward Bolle, Ber., **48**, 1770 (1916) *28*

CuS_5O_6

H_2O **III-1388**

An aqueous solution of cupric pentathionate crystallizes as small, blue prisms of tetrahydrated cupric pentathionate.

$$CuS_5O_6 + 4H_2O \rightarrow CuS_5O_6 \cdot (H_2O)_4$$

P. T. Cleve, Sv. Kem. Tidsk., **1**, 6 (1889) *10*

$CuSeO_3$

SeO_2 **III-1389**

H_2O

When normal copper selenite is digested with selenium dioxide in water at 60° and the residue recrystallized, anhydrous copper hydrogen selenite forms as blue-green, microscopic, four-sided prisms.

$$CuSeO_3 + SeO_2 + H_2O \rightarrow CuSeO_3 \cdot H_2SeO_3$$

L. F. Nilson, Nova Acta. Soc. Uppsal. [3], **9**, No. 7, 62 (1874) *10*

$CuSeO_4$

H_2S **III-1390**

Copper sulfide precipitates and selenic acid is formed when a suspension of copper selenate is treated with hydrogen sulfide.

$$CuSeO_4 + H_2S \rightarrow \underline{CuS} + H_2SeO_4$$

Wohlivill, Ann., **114**, 169 (1860)
Ref., L. M. Dennis and J. P. Koller, J. Am. Chem. Soc., **41**, 952 (1919) *1*

$CuSiF_6 \cdot 4H_2O$

NH_3 **III-1391**

Copper fluosilicate reacts with ammonia.

$$CuSiF_6 \cdot 4H_2O + 7NH_3 \rightarrow CuFOH \cdot 2NH_3 + 5NH_4F + SiO_2 + H_2O$$

L. Balbiano, Gazz. chim. ital., **14**, 81 (1884) *21*

$CuSiF_6 \cdot 6H_2O$

Δ **III-1392**

The decomposition of copper fluosilicate by means of a small amount of heat may be represented by the equation:

$$CuSiF_6 \cdot 6H_2O \rightarrow Cu(OH)F + SiF_4 + HF + 5H_2O$$

L. Balbiano, Gazz. chim. ital., **14**, 83 (1884) *21*

Cu-Sn-Sb

HCl **III-1393**

KCN

A rapid and accurate estimation of copper in Babbitt can be carried out by dissolving the alloy in hydrochloric acid, adding tartaric acid and a little nitric acid and ammonia, then titrating slowly with standard potassium cyanide.

(a) $(Cu + 2HCl \rightarrow CuCl_2 + H_2)$

(b) $(CuCl_2 + 4NH_4OH \rightarrow Cu(NH_3)_4Cl_2 + 4H_2O)$

(c) $(2Cu(NH_3)_4Cl_2 + 7KCN + H_2O$
$\rightarrow 2K_2Cu(CN)_3 + 2NH_4Cl + 2KCl + KOCN + 6NH_3)$

E. W. McHenry, Trans. Roy. Soc. (Can.) [III], **15**, 58 (1921) *69*

Cu

$(NH_4)_2S$ **III-1394**

Copper reacts with ammonium sulfide to give cuprous sulfide, ammonia and hydrogen.

$$(NH_4)_2S + 2Cu \rightarrow Cu_2S + 2NH_3 + H_2$$

Henmann, Gazz. chim. ital., **6**, 203 (1876) *21*

$Cu_2B_4O_7$

CuB_2O_4 **III-1395**

C

An electrolytic cell containing the K_2CO_3, Na_2CO_3, KCl eutectic with borax and copper oxide added and Cu screen electrodes has an EMF of 0.78 V. at 600°C. The total process is written as:

$$4CuB_2O_4 + C \rightarrow 2Cu_2B_4O_7 + CO_2.$$

S. Tamaru and M. Kamada, Z. Elektrochem., **41**, 95 (1935) *86*

Cu_2Br_2

NH_4Br **III-1396**

Upon cooling the mixture of a solution of cuprous bromide and ammonium bromide in presence of hydrobromic acid and copper wire, crystals are obtained.

(a) $$Cu_2Br_2 + 4NH_4Br \rightarrow (NH_4Br)_4Cu_2Br_2$$

(b) $$Cu_2Br_2 + 2NH_4Br + H_2O \rightarrow (NH_4Br)_2Cu_2Br_2 \cdot H_2O$$

H. L. Wells and E. B. Hurlburt, Z. anorg. Chem., **10**, 157 (1895) *28*

Cu_2Br_2

NH_4CNS **III-1397**

Cuprous thiocyanate diammoniate is precipitated as a white crystalline powder when ammonium thiocyanate is added to an ammoniacal cuprous bromide solution.

$$2NH_4CNS + Cu_2Br_2 + 2NH_3 \rightarrow Cu_2(CNS)_2 \cdot 2NH_3 + 2NH_4Br$$

Richards and Merigold, Proc. Am. Acad., **33**, 131–38
Ref., James Knox and A. B. Prescott, J. Am. Chem. Soc., **20**, 39 (1898) *1*

Cu_2Br_2

NH_4OH **III-1398**

Cuprous bromide diammoniate is prepared in long colorless prisms by dissolving cuprous bromide in the least possible amount of ammonium hydroxide and acetic acid, and evaporating over sulfuric acid in a current of hydrogen.

$$Cu_2Br_2 + 2NH_4OH \rightarrow Cu_2Br_2 \cdot 2NH_3 + (2H_2O)$$

T. W. Richards and B. S. Merigold, Proc. Am. Acad., **33**, 131–38
Ref., James Knox and A. B. Prescott, J. Am. Chem. Soc., **20**, 39 (1898) *1*

$Cu_2ClH_3O_3$

Δ **III-1399**

Atacamite decomposes when heated above 200°C to yield water vapor and a brown hygroscopic mixture of oxide and chloride of copper. When water is added to the mass the original compound is not regenerated. When heated above 250°, hydrochloric acid and water are given off and a mixture of oxide and chloride of copper remains.

(a) $2Cu_2ClO_3H_3 \rightarrow 3H_2O + CuCl_2 + 3CuO$

(b) $3Cu_2ClO_3H_3 \rightarrow 5CuO + CuCl_2 + HCl + 4H_2O$

E. Ludwig, Ann., **169**, 74 (1873) *50*

$Cu_2ClO_3H_3$

Δ **III-1400**

Atacamite on being heated to 220–250°C evolves water vapor, and leaves cupric chloride and cupric oxide as a residue.

$$2Cu_2ClO_3H_3 \rightarrow CuCl_2 + 3CuO + 3H_2O$$

E. Ludwig, Ber., **6**, 553 (1873) *11*

Cu_2Cl_2

CO **III-1401**

Carbon monoxide reduces cuprous chloride solution.

$$Cu_2Cl_2 + CO + H_2O \rightarrow \underline{2Cu} + CO_2 + 2HCl$$

Winkler, Z. anal. Chem., **28**, 272 (1889)
Ref., Jones, Am. Chem. J., **22**, 306 (1899) *1*

Cu_2Cl_2

Fe **III-1402**

Iron may be used to reduce cuprous chloride producing very pure copper.
Nails were used and the reaction proceeded equally well whether they were scoured or not. The copper obtained must be boiled with dilute HCl to remove any adhering particles.

$$3Cu_2Cl_2 + 2Fe \rightarrow \underline{6Cu} + (2FeCl_3)$$

E. Vigouroux, Bull. soc. chim. (France) [IV], **1**, 10 (1907) *31*

Cu_2Cl_2

NH_3 **III-1403**

When cuprous chloride is treated with ammonia gas at ordinary temperatures, the following addition compounds are formed. The same type compounds are formed with Cu_2Br_2 and Cu_2I_2.

(a) $Cu_2Cl_2 + NH_3 \rightarrow Cu_2Cl_2 \cdot NH_3$

(b) $Cu_2Cl_2 + 3NH_3 \rightarrow Cu_2Cl_2 \cdot 3NH_3$

(c) $Cu_2Cl_2 + 6NH_3 \rightarrow Cu_2Cl_2 \cdot 6NH_3$

S. J. Lloyd, J. Phys. Chem., **12**, 398 (1908) *7*

Cu_2Cl_2

NH_4Cl **III-1404**

Upon cooling the mixture of a solution of cuprous chloride and ammonium chloride in presence of hydrochloric acid and copper wire, colorless crystals are obtained.

(a) $Cu_2Cl_2 + 4NH_4Cl \rightarrow \underline{4NH_4Cl \cdot Cu_2Cl_2}$

(b) $3Cu_2Cl_2 + 4NH_4Cl \rightarrow \underline{4NH_4Cl \cdot 3Cu_2Cl_2}$

H. L. Wells and E. B. Hurlburt, Z. anorg. Chem., **10**, 157 (1895) *28*

Cu_2Cl_2

NO_2 **III-1405**

Cuprous chloride solution absorbs nitrogen dioxide with the formation of a color which is proportional in intensity to the amount of nitrogen dioxide absorbed. The equation is:

$$Cu_2Cl_2 + 2NO_2 \rightarrow [Cu_2(NO_2)_2]Cl_2$$

J. A. A. Auzies, Bull. soc. chim. (France), [4], **9**, 814 (1911) *31*

Cu_2Cl_2

NaOH **III-1406**

Cuprous chloride reacts with dilute sodium hydroxide to form orange-colored cuprous hydroxide in the cold (a). With hot caustic soda, red cuprous oxide is formed (b).

(a) $Cu_2Cl_2 + 2NaOH \rightarrow 2CuOH + 2NaCl$

(b) $Cu_2Cl_2 + 2NaOH \rightarrow Cu_2O + 2NaCl + H_2O$

W. D. Bancroft, Trans. Am. Electrochem. Soc., **8**, 46 (1905) *8*

Cu_2Cl_2

Na_2CO_3 **III-1407**

Solid cuprous chloride and solid sodium carbonate react by metathesis to a small extent, when shaken together.

$$Cu_2Cl_2 + Na_2CO_3 \rightarrow 2NaCl + Cu_2CO_3$$

H. G. Denham, J. Chem. Soc. (London), **113**, 405 (1918) *57*

Cu_2Cl_2

O_2 **III-1408**

If air is passed over hot cuprous chloride with moist sand above 100°C, Cu_2OCl_2 is formed. At a temperature of 400°C it decomposes giving off oxygen.

(a) $$2Cu_2Cl_2 + O_2 \rightarrow 2Cu_2OCl_2$$

(b) $$2Cu_2OCl_2 \rightarrow 2Cu_2Cl_2 + O_2$$

Mallet, Comp. rend., **64**, 226 (1867) *22*

Cu_2Cl_2

O_2 **III-1409**

Cupric chloride is formed when cuprous chloride reacts with oxygen in the presence of water.

(a) $$Cu_2Cl_2 + H_2O \rightarrow Cu_2O + 2HCl$$

(b) $$2Cu_2Cl_2 + 4HCl + O_2 \rightarrow 4CuCl_2 + 2H_2O$$

M. Gröger, Z. anorg. Chem., **28**, 154 (1901) *28*

Cu_2Cl_2

O_2 **III-1410**

If cuprous chloride is exposed to air the following reaction takes place.

$$4Cu_2Cl_2 + O_2 \rightarrow 2Cu_2O + 4CuCl_2$$

B. Lean and W. H. Whatmough, J. Chem. Soc. (London), **73**, 150 (1898) *103*

Cu_2Cl_2

III-1411

O_2

HCl

When air is blown through a 10% solution of cuprous chloride in sodium chloride solution, cupric chloride is regenerated easily. Stronger solutions do not regenerate so easily.

(a) $2Cu_2Cl_2 + O_2 \rightarrow 2CuCl_2 + 2CuO$

(b) $2CuO + 4HCl \rightarrow 2CuCl_2 + 2H_2O$

F. Fischer and P. Dilthey, Brenn. Chem., **7**, 300 (1926) *11*

$Cu_2Cl_2(CO)_2 \cdot 4H_2O$

P

III-1412

Under pressure dicarbonyl cuprous chloride decomposes slightly, according to the equations:

(a) $Cu_2Cl_2(CO)_2 \cdot 4H_2O \rightarrow Cu_2Cl_2 + 2CO + 4H_2O$

(b) $Cu_2Cl_2 + CO \rightarrow 2Cu + COCl_2$

(c) $COCl_2 + H_2O \rightarrow 2HCl + CO_2$

W. A. Jones, Am. Chem. J., **22**, 301 (1899) *13*

$(CuCl)_2C_2H_5SCH_2COOH \cdot H_2O$

$C_2H_5SCH_2COOH$

III-1413

KI

Hydrated ethylthioglycolic acid cuprous chloride is dissolved in ethylthioglycolic acid and water and potassium iodide solution added drop by drop. A heavy, colorless oil separates which soon forms small crystals of anhydrous ethylthioglycolic acid cuprous iodide.

$$(CuCl)_2 \cdot C_2H_5SCH_2COOH \cdot H_2O + C_2H_5SCH_2COOH + 2KI \rightarrow (CuI)_2(C_2H_5SCH_2COOH)_2 + (H_2O + 2KCl)$$

L. Ramber, Acta Univ. Lund. Med. Matem. Natur., N. S., **4**, No. 2, 13 (1908) *10*

$Cu_2Cl_2 \cdot 2NH_3$

C_2H_2 **III-1414**

H_2O

Cuprous chloride diammoniate reacts with acetylene and water to yield acetylene cuprous oxide and ammonium chloride.

$$Cu_2Cl_2 \cdot 2NH_3 + H_2O + C_2H_2 \rightarrow C_2H_2Cu_2O + 2NH_4Cl$$

Blochman, Gazz. chim. ital., **4**, 298 (1874) *21*

$Cu_2(CN)_2$

KCN **III-1415**

Cuprous cyanide combines with alkali cyanides, giving double salts.

$$2Cu_2(CN)_2 + KCN \rightarrow KCN \cdot 2Cu_2(CN)_2$$

A. Lallemand, Compt. rend., **58**, 750 (1864) *29*

$Cu_2(CN)_2$

N_2O **III-1416**

At moderate temperatures nitrous oxide reduces cuprous cyanide to metallic copper, carbon dioxide and nitrogen.

$$Cu_2(CN)_2 + 4N_2O \rightarrow 2Cu + 2CO_2 + 5N_2$$

A. C. Vournasos, Bull. soc. chim. (France) [4], **21**, 286 (1917) *31*

Cu_2CO_3

CrO_3 **III-1417**

10 gms. of cuprous carbonate and 26 gms. of chromium trioxide are dissolved in a little water and the solution made up to 200 cc. Concentrated ammonia solution is added until a clear liquid is obtained. The double chromate separates out in large shining black crystals.

$$Cu_2CO_3 + 4CrO_3 + 8NH_4OH \rightarrow 2[(NH_4)_2Cu(CrO_4)_2 \cdot 2NH_3] + 6H_2O + CO$$

S. H. C. Briggs, J. Chem. Soc. (London), **83**, 394 (1903) *102*

Cu_2C_2

III-1418

H_2

Ethylene is obtained when copper acetylide is treated with hydrogen, the latter being produced by the action of zinc on ammonium hydroxide.

$$Cu_2C_2 + 2H_2 \rightarrow C_2H_4 + 2Cu$$

Bertholet, Comp. rend., **50**, 808 (1860) *29*
Ref., Bertholet, Comp. rend., **54**, 516 (1862)

$Cu_2C_2 \cdot H_2O$

III-1419

HCl

Mineral acids decompose cuprous acetylide monohydrate to form water, acetylene and cuprous chloride.

$$Cu_2C_2 \cdot H_2O + 2HCl \rightarrow C_2H_2 + Cu_2Cl_2 + H_2O$$

R. Blochmann, Ber., **7**, 275 (1874) *11*

$(Cu_2(C_5H_5N)_4)Cl_2$

III-1420

Cl_2

Chlorine passed into aqueous solutions of $(Cu_2(C_5H_5N)_4)Cl_2$ slowly transforms copper into the divalent form, yielding a green-yellow complex.

$$2(Cu_2(C_5H_5N)_4)Cl_2 + 2Cl_2 \rightarrow 2(Cu(C_5H_5N)_4)Cl_2 + 2CuCl_2$$

D. O. Marouhian, J. Gen. Chem. (USSR), **10**, 918 (1940) *60*

$Cu_2Fe(CN)_6$

III-1421

$H_2C_2O_4$

A green-yellow precipitate is formed when copper ferrocyanide is boiled with 5% oxalic acid for two hours with a reflux condenser.

$$Cu_2Fe(CN)_6 + 3H_2C_2O_4 \rightarrow \underline{2CuC_2O_4} + \underline{FeC_2O_4} + 6HCN$$

Leuba, Ann. de chim. appl., **10**, 218
Ref., J. Am. Chem. Soc., **28**, 1771 (1906) *1*

$Cu_2Fe(CN)_6$

H_2O **III-1422**

Cupric ferrocyanide, cupric ferricyanide, Prussian and Turnbull's blues do *not* form stable hydrates at ordinary temperature. Vapor pressure vs. composition curves demonstate the absence of stable compounds. This conclusion is contrary to claims by Wyrouboff (Ann. chim. phys. [5], **8**, 44 (1876)) and many others.

(a) $Cu_2Fe(CN)_6 + H_2O \rightarrow Cu_2Fe(CN)_6 \cdot H_2O$

(b) $Cu_3[Fe(CN)_6]_2 + H_2O \rightarrow Cu_3[Fe(CN)_6]_2 \cdot H_2O$

(c) $Fe_4[Fe(CN)_6]_3 + H_2O \rightarrow Fe_4[Fe(CN)_6]_3 \cdot H_2O$

(d) $Fe_3[Fe(CN)_6]_2 + H_2O \rightarrow Fe_3[Fe(CN)_6]_2 \cdot H_2O$

H. B. Weiser, W. O. Milligan and J. B. Bates, J. Phys. Chem., **45**, 701 (1941) *7*

$Cu_2Fe(CN)_6$

$K_2C_2O_4$ **III-1423**

Cupric ferrocyanide reacts with potassium oxalate to form copper oxalate and potassium ferrocyanide.

$$Cu_2Fe(CN)_6 + 2K_2C_2O_4 \rightarrow 2CuC_2O_4 + K_4Fe(CN)_6$$

M. Kohn, Z. anorg. Chem., **197**, 287 (1931) *4*

$Cu_2Fe(CN)_6$

$Li_4Fe(CN)_6$ **III-1424**

Red crystals are formed by boiling cupric ferrocyanide with lithium ferrocyanide.

$$Cu_2Fe(CN)_6 + Li_4Fe(CN)_6 \rightarrow 2Li_2CuFe(CN)_6$$

J. Messner, Z. anorg. Chem., **9**, 126 (1895) *28*

$Cu_2Fe(CN)_6$

III-1425

$Mg_2Fe(CN)_6$

By boiling cupric ferrocyanide with a solution of magnesium ferrocyanide solution brown colored crystals of magnesium cupric ferrocyanide are obtained. The same type reaction occurs with calcium ferrocyanide, sodium ferrocyanide, strontium ferrocyanide, barium ferrocyanide.

(a) $Cu_2Fe(CN)_6 + Mg_2Fe(CN)_6 \rightarrow \underline{2MgCuFe(CN)_6}$

(b) $Cu_2Fe(CN)_6 + Ca_2Fe(CN)_6 \rightarrow \underline{2CaCuFe(CN)_6}$

(c) $Cu_2Fe(CN)_6 + Na_4Fe(CN)_6 \rightarrow \underline{2Na_2CuFe(CN)_6}$

(d) $Cu_2Fe(CN)_6 + Sr_2Fe(CN)_6 \rightarrow \underline{2SrCuFe(CN)_6}$

(e) $Cu_2Fe(CN)_6 + Ba_2Fe(CN)_6 \rightarrow \underline{2BaCuFe(CN)_6}$

J. Messner, Z. anorg. Chem., **8**, 368 (1895) *28*

$Cu_2Fe(CN)_6$

III-1426

$(NH_4)_4Fe(CN)_6$

Red brown colored crystals of ammonium cupric ferrocyanide are obtained by boiling cuprous ferrocyanide with a solution of ammonium ferrocyanide.

$$Cu_2Fe(CN)_6 + (NH_4)_4Fe(CN)_6 \rightarrow \underline{2(NH_4)_2CuFe(CN)_6}$$

J. Messner, Z. anorg. Chem., **8**, 368 (1895) *28*

Cu_2HIO_6

III-1427

Δ

By heating cupric acid orthoperiodate to 115°, the product will be cupric orthodiperiodate and water.

$$2Cu_2HIO_6 \rightarrow Cu_4I_2O_{11} + H_2O$$

F. Giolitti, Gazz. chim. ital., **32**, 351 (1902) *21*

Cu_2I_2

III-1428

NH_4I

Upon cooling the mixture of a solution of cuprous iodide and ammonium iodide, colorless crystals are obtained.

$$Cu_2I_2 + 2NH_4I \rightarrow (NH_4I)_2Cu_2I_2$$

H. L. Wells and E. B. Hurlburt, Z. anorg. Chem., **10**, 157 (1895) *28*

$Cu_2(NH_3)_2CO_3$

CO **III-1429**

Carbon monoxide acts on a solution of ammoniacal cuprous carbonate to form ammonium carbonate and copper.

$$Cu_2(NH_3)_2CO_3 + CO + 2NH_3 + 2H_2O \rightarrow 2Cu + 2(NH_4)_2CO_3$$

A. T. Larson and C. S. Tertswort, J. Am. Chem. Soc., **44**, 2884 (1922) *13*

$Cu_2(NH_3)_4CO_3$

CO **III-1430**

At temperatures above 60°C copper is reduced to the metallic state when cuprous ammonium carbonate comes in contact with carbon monoxide.

$$Cu_2(NH_3)_4CO_3 + CO + 2H_2O \rightarrow 2(NH_4)_2CO_3 + 2Cu$$

W. Gump and I. Ernst, Ind. Eng. Chem., **22**, 382 (1930) *24*

Cu_2O

$AgNO_3$ **III-1431**

Metallic silver is obtained by the reaction of cuprous oxide with silver nitrate.

$$3Cu_2O + 6AgNO_3 + 3H_2O \rightarrow 2Cu_2H_3NO_6 + 2CuN_2O_6 + 6Ag$$

W. Hampe, Z. anal. Chem., **17**, 129 (1878) *28*

Cu_2O

BaO_2 **III-1432**

When a mixture of cuprous oxide and barium peroxide is heated black cupric oxide is formed.

$$Cu_2O + BaO_2 \rightarrow 2CuO + BaO$$

J. A. Hedvall and N. von Zweigbergk, Ark. Kem., Min. Geol., **7**, Part 20, 4 (1919) *10*

Cu_2O

III-1433

$(CH_3)_2SO_4$

Pure cuprous sulfate is formed when cuprous oxide reacts with dimethyl sulfate at 160°.

$$Cu_2O + (CH_3)_2SO_4 \rightarrow Cu_2SO_4 + (CH_3)_2O$$

Recoura, Compt. rend., **148**, 1105 (1909) *1*
Ref., J. L. Howe, J. Am. Chem. Soc., **31**, 1293 (1909)

Cu_2O

III-1434

CuP

In the production of copper ruby glass copper phosphide may be used with cuprous oxide.

$$5Cu_2O + 2CuP \rightarrow 12Cu + P_2O_5$$

W. A. Weyl and N. J. Kreidl, J. Am. Cer. Soc., **24**, 339 (1941) *16*

Cu_2O

III-1435

Cu_2S

Cuprous oxide reacts with copper sulfide to form metallic copper.

$$2Cu_2O + Cu_2S \rightarrow 6Cu + SO_2$$

W. A. Weyl and N. J. Kreidl, J. Am. Cer. Soc., **24**, 339 (1941) *16*

Cu_2O

III-1436

HF

Cuprous oxide will react with hydrofluoric acid yielding cupric fluoride and metallic copper.

$$Cu_2O + 2HF \rightarrow CuF_2 + \underline{Cu} + H_2O$$

A. Jaeger, Z. anorg. Chem., **27**, 22 (1901) *28*

Cu_2O

III-1437

HN_3

Cuprous oxide reduces triazoic acid to ammonia and is converted into cupric triazoate in presence of excess acid.

$$Cu_2O + 5HN_3 \rightarrow 2CuN_6 + H_2O + NH_3 + N_2$$

J. W. Turrentine and R. L. Moore, J. Am. Chem. Soc., **34**, 380 (1912) *1*

Cu_2O

$K_2Cr_2O_7$ **III-1438**

H_2SO_4

Cuprous oxide is oxidized by potassium dichromate in the presence of dilute sulfuric acid.

$$3Cu_2O + K_2Cr_2O_7 + 10H_2SO_4 \rightarrow 6CuSO_4 + K_2SO_4 + 10H_2O + Cr_2(SO_4)_3$$

B. B. Ross, J. Am. Chem. Soc., **18**, 922 (1896) *1*

Cu_2O

$MgCl_2$ **III-1439**

Cuprous oxide dissolves in a hot solution of magnesium chloride with the formation of magnesium hydroxide and cuprous chloride.

$$Cu_2O + H_2O + MgCl_2 \rightarrow 2CuCl + Mg(OH)_2$$

Sterry Hunt,
Ref., Ch. Friedel, Ber., **3**, 35 (1870) *11*

Cu_2O

NH_2OH **III-1440**

Cuprous oxide is reduced to metallic copper when boiled with an excess of hydroxylamine.

$$Cu_2O + 2NH_2OH \rightarrow \underline{2Cu} + 3H_2O + N_2$$

M. Adams and E. Overman, J. Am. Chem. Soc., **31**, 640 (1909) *1*

Cu_2O

$(NH_4)_2SO_4$ **III-1441**

Operating in an atmosphere of hydrogen, cuprous oxide and ammonium sulfate are dissolved in aqueous ammonia and the cuprous sulfate tetrammoniate formed is precipitated at 50° by adding boiled alcohol.

$$Cu_2O + (NH_4)_2SO_4 + 2NH_4OH \rightarrow \underline{Cu_2SO_4 \cdot 4NH_3} + 3H_2O$$

Bouzat, Compt. rend., **146**, 76 (1908) *38*

Cu_2O

III-1442

$(NH_4)_2SO_4$

NH_3

Ammonio-cuprous sulfate is precipitated as a white crystalline powder on addition of ethyl alcohol to a solution of cuprous oxide and ammonium sulfate in aqueous ammonia at 50° in an atmosphere of hydrogen.

$$Cu_2O + (NH_4)_2SO_4 + 2NH_3 \rightarrow \underline{Cu_2SO_4 \cdot 4NH_3} + (H_2O)$$

A. Bouzat, Compt. rend., **146**, 75 (1908)
Ref., M. A. Whiteley, J. Chem. Soc. (London), **94:2**, 187 (1908) *57*

Cu_2O

III-1443

$SOCl_2$

Cupric chloride is formed when cuprous chloride is heated with thionyl chloride in a sealed tube at 200°.

$$6CuCl + 4SOCl_2 \rightarrow 6CuCl_2 + S_2Cl_2 + 2SO_2$$

H. B. North and A. M. Hageman, J. Am. Chem. Soc., **35**, 356 (1913) *1*

Cu_2O

III-1444

Δ

On heating, cuprous oxide decomposes into cupric oxide and metallic copper.

$$Cu_2O \rightarrow CuO + Cu$$

W. A. Weyl and N. J. Kreidl, J. Am. Cer. Soc., **24**, 339 (1941) *16*

$Cu_2OSO_4 \cdot 2NH_2OH$

III-1445

NaOH

Cuprous oxide forms when a water solution of sodium hydroxide reacts with a solution containing $Cu_2OSO_4 \cdot 2NH_2OH$.

$$Cu_2OSO_4 \cdot 2NH_2OH + 2NaOH \rightarrow \underline{Cu_2O} + Na_2SO_4 + 4H_2O + N_2$$

M. Adams and E. Overman, J. Am. Chem. Soc., **31**, 640 (1909) *1*

$Cu_2P_2O_7$

$K_4P_2O_7$ **III-1446**

Copper pyrophosphate dissolves in a solution of potassium pyrophosphate. Evaporation of the resulting solution either at ordinary temperature or on the water bath produces amorphous, light blue, tetra-hydrated double pyrophosphate of copper and potassium.

$$3Cu_2P_2O_7 + 3K_4P_2O_7 + 4H_2O \rightarrow (Cu_2P_2O_7 \cdot K_4P_2O_7)_3 \cdot 4H_2O$$

C. N. Pahl, K. Sven. Vet. Ak. Handl. Övers., **30**, No. 7, 44 (1873) *10*

$Cu_2P_2O_7$

$Na_4P_2O_7$ **III-1447**

Copper pyrophosphate dissolves in a solution of sodium pyrophosphate. Cooling yields pale blue, microscopic, rhombic tablets of 3:1:21 double pyrophosphate. The resulting mother liquor, evaporated at about 40° yields a light blue 1:3:4 double pyrophosphate crystals. The next mother liquor yield the anhydrous double pyrophosphate as blue, large triclinic prisms.

(a) $3Cu_2P_2O_7 + Na_4P_2O_7 + 21H_2O \rightarrow (Cu_2P_2O_7)_3 \cdot Na_4P_2O_7 \cdot (H_2O)_{21}$

(b) $Cu_2P_2O_7 + 3Na_4P_2O_7 + 4H_2O \rightarrow Cu_2P_2O_7 \cdot (Na_4P_2O_7)_3 \cdot (H_2O)_4$

(c) $Cu_2P_2O_7 + 3Na_4P_2O_7 \rightarrow Cu_2P_2O_7 \cdot (Na_4P_2O_7)_3$

C. N. Pahl, K. Sven. Vet. Ak. Handl. Övers., **30**, No. 7, 43 (1873) *10*

$(Cu_2P_2O_7)_2 \cdot Na_4P_2O_7$

Δ **III-1448**

When the double pyrophosphate of copper and sodium is kept at about 100° it decomposes and yields basic copper pyrophosphate as a pretty bluish-green, crystalline salt.

$$(Cu_2P_2O_7)_2 \cdot Na_4P_2O_7 + 7H_2O \rightarrow Na_4P_2O_7 + Cu_2P_2O_7 \cdot (Cu(OH)_2)_2 \cdot (H_2O)_3 + H_4P_2O_7$$

C. N. Pahl, K. Sven. Vet. Ak. Handl. Övers., **30**, No. 7, 41 (1873) *10*

Cu_2S

III-1449

$AgNO_3$

Cuprous sulfide in contact with silver nitrate solution forms cupric nitrate, silver sulfide and silver.

$$Cu_2S + 4AgNO_3 \rightarrow 2Cu(NO_3)_2 + Ag_2S + 2Ag$$

Karl Heumann, Ber., **7**, 1680 (1874) *11*

Cu_2S and CuS

III-1450

$AgNO_3$

In neutral solution, silver nitrate reacts with Copper I and II sulfides according to equations (a) and (b).
Treatment of the precipitate with ferric nitrate (6%) removes silver as $AgNO_3$.

(a) $$Cu_2S + 4AgNO_3 \rightarrow 2Cu(NO_3)_2 + \underline{Ag_2S + 2Ag}$$

(b) $$CuS + 2AgNO_3 \rightarrow \underline{Ag_2S} + Cu(NO_3)_2$$

(c) $$Ag + Fe(NO_3)_3 \rightarrow AgNO_3 + Fe(NO_3)_2$$

J. Hanus and V. Horvorka, Coll. Czech. Comm., **1**, 65 (1929) *87*

Cu_2S

III-1451

$Cu_2Fe_2O_4$

$Cu_2Mn_2O_4$

Copper sulfide reacts with copper ferrite and manganite at 700°C, and is reduced to the metal.

(a) $$3Cu_2Fe_2O_4 + 2Cu_2S \rightleftharpoons 10Cu + 2Fe_3O_4 + 2SO_2$$

(b) $$3Cu_2Mn_2O_4 + 2Cu_2S \rightleftharpoons 10Cu + 2Mn_3O_4 + 2SO_2$$

R. Schenk and H. Keuth, Z. Elektrochem., **46**, 303, 304 (1940) *86*

Cu_2S

III-1452

Cu_2O

The reversible copper roasting process at 700°C and the SO_2 pressure is markedly influenced by foreign materials which react with the solid phase.

$$2Cu_2O + Cu_2S \rightleftharpoons 6Cu + SO_2.$$

R. Schenk and H. Keuth, Z. Elektrochem., **46**, 298, 308 (1940) *86*

Cu_2S

Cu_2O **III-1453**

A mixture of cuprous sulfide and cuprous oxide reacts at high temperature and yields copper and sulfur dioxide. Cuprous sulfide and ferrous sulfide under similar conditions yield copper but not sulfur dioxide.

(a) $$Cu_2S + 2Cu_2O \rightarrow 6Cu + SO_2$$

(b) $$Cu_2S + FeS \rightarrow 2Cu + FeS_2$$

J. H. L. Vogt, Vid. sel. ch. Skrifter., **1**, 27 (1918) *10*

Cu_2S

$FeCl_3$ **III-1454**

Chalcocite is readily attacked by ferric chloride producing cupric sulfide.

$$Cu_2S + 2FeCl_3 \rightarrow \underline{CuS} + CuCl_2 + 2FeCl_2$$

H. N. Stokes, J. Am. Chem. Soc., **29**, 314 (1907) *1*

Cu_2S

$Fe_2(SO_4)_3$ **III-1455**

Cuprous sulfide is dissolved by ferric sulfate solutions to form copper sulfate, ferrous sulfate and sulfur.

$$Cu_2S + 2Fe_2(SO_4)_3 \rightarrow 2CuSO_4 + 4FeSO_4 + S$$

N. S. Keith, Trans. Am. Electrochem. Soc., **1**, 137 (1902) *8*

Cu_2S

$Fe_2(SO_4)_3$ **III-1456**

A suspension of copper sulfide goes into solution during electrolysis in the presence of ferric sulfate and free sulfuric acid.

$$Cu_2S + 2Fe_2(SO_4)_3 + xH_2SO_4 \rightarrow 2CuSO_4 + 4FeSO_4 + xH_2SO_4$$

E. Cohen, Z. Elektrochem., **1**, 53 (1894) *86*

Cu_2S

III-1457

$Fe_2(SO_4)_3$

The reactions that take place in the process for the extraction of copper from pyritic ores with ferric sulfate solution as the leaching reagent are:

(a) $Cu_2S + Fe_2(SO_4)_3 \rightarrow CuSO_4 + 2FeSO_4 + CuS$

(b) $CuS + 4Fe_2(SO_4)_3 + 4H_2O \rightarrow CuSO_4 + 8FeSO_4 + 4H_2SO_4$

W. D. Horne and E. W. Rice, Ind. Eng. Chem., **15**, 1271 (1923) *23*

Cu_2S

III-1458

HCN

A process of cyanide recovery from coke manufacture makes use of copper compounds to extract the cyanide from the gas instead of the iron salts that are usually employed.

(a) $Cu_2S + 6HCN + 4NH_3 \rightarrow 2(NH_4)_2Cu(CN)_3 + H_2S$

(b) $CuCN + 2HCN + 2NH_3 \rightarrow (NH_4)_2Cu(CN)_3$

(c) $(NH_4)_2Cu(CN)_3 + H_2SO_4 \rightarrow (NH_4)_2SO_4 + CuCN + 2HCN$

(d) $2CuCN + H_2S \rightarrow Cu_2S + 2HCN$

(e) $(NH_4)_2Cu(CN)_3 \rightarrow NH_4Cu(CN)_2 + NH_3 + HCN$

M. E. Mueller, Chem. & Met. Eng., **30**, 978 (1924) *44*

Cu_2S

III-1459

HI

Cuprous sulfide (chalcocite) reacts with hydriodic acid to form cuprous iodide and hydrogen sulfide.

$$Cu_2S + 2HI \rightarrow 2CuI + H_2S$$

Ed. Meusel, Ber., **3**, 123 (1870) *11*

Cu_2S

III-1460

H_2SO_4

Cuprous sulfide is oxidized to free sulfur when boiled with concentrated sulfuric acid.

(a) $Cu_2S + 2H_2SO_4 \rightarrow CuS + CuSO_4 + SO_2 + 2H_2O$

(b) $CuS + 2H_2SO_4 \rightarrow \underline{S} + CuSO_4 + SO_2 + 2H_2O$

C. Baskerville, J. Am. Chem. Soc., **17**, 910 (1895) *1*

Cu_2S

$(NH_4)_2S \cdot Sx$ **III-1461**

Yellow ammonium sulfide reacts with cuprous sulfide to form cupric sulfide.

$$Cu_2S + (NH_4)_2S \cdot Sx \rightarrow 2CuS + (NH_4)_2S \cdot Sx - 1$$

Karl Heumann, Ber, **6**, 752 (1873) *11*

Cu_2S

O_2 **III-1462**

Cuprous sulfide heated in the presence of air yields cupric sulfate and cupric oxide.

$$2Cu_2S + 5O_2 \rightarrow 2CuSO_4 + 2CuO$$

P. Klason, Sv. Kem. Tidsk., **29**, 192 (1917) *10*

Cu_2SO_4

CH_3COOH **III-1463**

O_2

Glacial acetic acid dissolves small quantities of cuprous sulfate to form an intensely violet solution which oxidizes rapidly in air, turning blue and finally depositing crystals of acid cupric acetate.

$$Cu_2SO_4 + 4CH_3CO_2H + \tfrac{1}{2}O_2 \rightarrow 2Cu(CH_3CO_2)_2 + H_2SO_4 + H_2O$$

A. Recoura, Compt. rend., **148**, 1108 (1909) *38*

Cu_2SO_4

H_2O **III-1464**

Cuprous sulfate is decomposed in presence of water to form cupric sulfate and metallic copper.

$$Cu_2SO_4 \rightarrow CuSO_4 + \underline{Cu}$$

J. T. Cundall, J. Chem. Soc. (London), **105**, 61 (1914) *57*
Ref., Recoura, Compt. rend., **148**, 1105 (1909) *38*

Cu_2SO_4

NaHS **III-1465**

A solution of cuprous sulfate is treated with sodium hydrogen sulfide and heated to about 80°. Powdered cupric sulfide is precipitated.

$$Cu_2SO_4 + 2NaHS \rightarrow \underline{Cu_2S} + (Na_2SO_4 + H_2S)$$

J. Girard, Ann. chim. anal., **4**, 383 (1899) *76*

Cu_2SO_4

NaOH **III-1466**

Copper sulfate reacts in the cold with dilute sodium hydroxide to form bluish cupric hydroxide. When the solutions are hot black cupric oxide is formed.

(a) Cold: $CuSO_4 + 2NaOH \rightarrow Cu(OH)_2 + Na_2SO_4$

(b) Hot: $CuSO_4 + 2NaOH \rightarrow CuO + Na_2SO_4 + H_2O$

W. D. Bancroft, Trans. Am. Electrochem. Soc., **8**, 46 (1905) *8*

Cu_2SO_4

O_2 **III-1467**

Dry cuprous sulfate oxidizes slowly in dry air at room temperature. At 200°C it fixes one atom of oxygen and is transformed into a brownish black mixture of cupric oxide and cupric sulfate.

$$Cu_2SO_4 + \tfrac{1}{2}O_2 \rightarrow CuO + CuSO_4$$

A. Recoura, Compt. rend., **148**, 1108 (1909) *38*

Cu_2SO_4

O_2 **III-1468**

If the copper voltameter is exposed to air oxygen, which oxidizes cuprous ions to cupric, cupric sulfate is formed in acid solution, whereas the basic sulfate is formed in neutral solution.

(a) $2Cu_2SO_4 + 2H_2SO_4 + O_2 \rightarrow 4CuSO_4 + 2H_2O$

(b) $2Cu_2SO_4 + 2H_2O + O_2 \rightarrow 2Cu(OH)_2 \cdot CuSO_4$

F. Förster, Z. Elektrochem., **3**, 481 (1897) *86*

$Cu_2SO_4 \cdot 4NH_3$

HNO_3 **III-1469**

Cuprous sulfate tetrammoniate reduces nitric acid with evolution of brown vapors.

$$Cu_2SO_4 \cdot 4NH_3 + 8HNO_3 \rightarrow 2NO_2 + (CuSO_4 + Cu(NO_3)_2 + 4NH_4NO_3 + 2H_2O)$$

Bouzat, Compt. rend., **146**, 77 (1908) *38*

$Cu_2SO_4 \cdot 4NH_3$

H_2O **III-1470**

Cuprous sulfate tetrammoniate is decomposed by water with the formation of a precipitate of cuprous oxide.

$$Cu_2SO_4 \cdot 4NH_3 + 3H_2O \rightarrow \underline{Cu_2O} + ((NH_4)_2SO_4 + 2NH_4OH)$$

Bouzat, Compt. rend., **146**, 77 (1908) *38*

$Cu_2SO_4 \cdot 4NH_3$

H_2SO_4 **III-1471**

Cuprous sulfate tetrammoniate treated with dilute sulfuric acid, gives a precipitate of copper and a solution of cupric sulfate and ammonium sulfate.

$$Cu_2SO_4 \cdot 4NH_3 + 2H_2SO_4 \rightarrow \underline{Cu} + CuSO_4 + 2(NH_4)_2SO_4$$

Bouzat, Compt. rend., **146**, 77 (1908) *38*

$Cu_2S_2O_3 \cdot 2(NH_4)_2S_2O_3$

$BaCl_2$ **III-1472**

A solution of copper ammonium thiosulfate will react with a concentrated solution of barium chloride yielding a white, amorphous precipitate of copper barium thiosulfate.

$$Cu_2S_2O_3 \cdot 2(NH_4)_2S_2O_3 + 2BaCl_2 + 4H_2O \rightarrow \underline{Cu_2S_2O_3 \cdot (BaS_2O_3)_2 \cdot 4H_2O} + 4NH_4Cl$$

A. Rosenheim and S. Steinhäuser, Z. anorg. Chem., **25**, 72 (1900) *28*

$Cu_2S_4O_6$

III-1473

Δ

Cuprous tetrathionate is unstable, even in the cold; on warming it is transformed into copper sulfide and sulfur trioxide.

$$Cu_2S_4O_6 \rightarrow 2CuS + 2SO_3$$

G. Chancel and E. Diacon, Compt. rend., **56**, 711 (1863) *29*

Cu_2S_6

III-1474

Δ

By gently heating copper hexasulfide, sulfur is given off and cupric trisulfide in amorphous form is obtained.

$$Cu_2S_6 \rightarrow Cu_2S_3 + 3S$$

A. Rössing, Z. anorg. Chem., **25**, 407 (1900) *28*

Cu_2Se

KCN **III-1475**

H_2O

When cuprous selenide is treated with an aqueous solution of potassium cyanide in the presence of air, the products are potassium copper cyanide, potassium selenium cyanide, and potassium hydroxide.

$$2Cu_2Se + 10KCN + O_2 + 2H_2O \rightarrow 2K_2Cu_2(CN)_4 + 2KSeCN + 4KOH$$

J. Landin, Sv. Kem. Tidsk., **12**, 2 (1900) *10*

Cu_2Si

S **III-1476**

Free silicon is formed when pulverized copper silicide is heated in closed tubes at 200–250°C, but when the temperature rises to 300°, the free silicon reacts with sulfur.

(a) $$Cu_2Si + S \rightarrow Cu_2S + Si$$

(b) $$Si + S_2 \rightarrow SiS_2$$

G. De Chalmot, Am. Chem. J., **19**, 871 (1897)
Ref., J. Am. Chem. Soc., **20**, 40 (1898) *1*

Cu_2Si

Zn **III-1477**

Free silicon is formed when pulverized copper silicide is allowed to react with zinc at a high temperature.

$$Cu_2Si + Zn \rightarrow ZnCu_2 + Si$$

G. De Chalmot, Am. Chem. J., **20**, 437 (1898)
Ref., J. Am. Chem. Soc., **20**, 111 (1898) *1*

$Cu_3(C_6H_5O_7)_2$

K_2CO_3 **III-1478**

$K_3C_6H_5O_7$

When a concentrated solution of potassium carbonate is added to copper citrate and potassium citrate, some carbon dioxide is evolved, and on evaporation, a complex potassium cupricitrate is obtained in which the copper is electronegative. The by-product of formation of carbon dioxide is not determined. Alkali hydroxide precipitates basic copper citrate from solution of this compound.

$$3Cu_3(C_6H_5O_7)_2 + 6K_3C_6H_5O_7 + 6K_2CO_3 \rightarrow 3K_{10}Cu_3(C_6H_5O_7)_4(CO_3)_2$$

S. U. Pickering, J. Chem. Soc. (London), **97**, 1844, 1948 (1910) *57*

$Cu_3(C_6H_5O_7)_2 \cdot CuO \cdot 2H_2O$

$K_3C_6H_5O_7 \cdot H_2O$ **III-1479**

When basic copper citrate is added to an aqueous solution of potassium citrate containing citric acid, heated on a water bath, a precipitate of potassium cupricitrate is formed. In this compound the copper is part of a complex electronegative radical.

$$3[Cu_3(C_6H_5O_7)_2 \cdot CuO \cdot 2H_2O] + 4[K_3C_6H_5O_7 \cdot H_2O] + 2H_3C_6H_5O_7 \rightarrow 3[K_4H_2(C_6H_5O_7)_2 \cdot CuO] + (3Cu_3(C_6H_5O_7)_2 + 10H_2O)$$

S. U. Pickering, J. Chem. Soc. (London), **97**, 1841 (1910) *57*

$Cu_3Co_2(CN)_{12}$

III-1480

NH_4OH

Small shining blue crystals of $Cu_3Co_2(CN)_{12} \cdot 2NH_3 \cdot 5H_2O$ are obtained when cupri cobalticyanide is treated with ammonium hydroxide.

$$Cu_3Co_2(CN)_{12} + 2NH_4OH + 3H_2O \rightarrow Cu_3Co_2(CN)_{12} \cdot 2NH_3 \cdot 5H_2O$$

Constantin Zwenger, Ann., **62**, 158 (1847)
Ref., E. H. Miller and J. A. Mathews, J. Am. Chem. Soc., **22**, 64 (1900) *1*

Cu_3PS_4

III-1481

Δ

Cuprous sulfide is obtained by very strong heating of cuprous thiophosphate.

$$2Cu_3PS_4 \rightarrow \underline{3Cu_2S} + P_2S_5$$

E. Glatzel, Z. anorg. Chem., **4**, 186 (1893) *28*

$Cu_4H_6O_{10}S$

III-1482

Δ

Brochantite decomposes above 300°C to yield water vapor, copper sulfate, and copper oxide.

$$Cu_4H_6O_{10}S \rightarrow CuSO_4 + 3CuO + 3H_2O$$

E. Ludwig, Ann., **169**, 79 (1873) *50*

$Cu_5(P_3O_{10})_2$

III-1483

H_2S

Triphosphoric acid is obtained by the reaction of copper triphosphate with hydrogen sulfide.

$$2Cu_5(P_3O_{10})_2 + 10H_2S \rightarrow 4H_5P_3O_{10} + 10CuS$$

F. Schwarz, Z. anorg. Chem., **9**, 249 (1895) *28*

$(HCOO)_2Cu$

H_2O **III-1484**

When solutions of copper formate are boiled, an insoluble pale green basic salt is formed. The same compound is formed by addition of copper hydroxide to copper formate. It is converted to a less basic salt on contact with normal copper formate.

(a) $4(HCOO)_2Cu + 6H_2O \rightarrow \underline{(HCOO)_2Cu \cdot 3Cu(OH)_2} + (6HCOOH)$

(b) $(HCOO)_2Cu + 3Cu(OH)_2 \rightarrow \underline{(HCOO)_2Cu \cdot 3Cu(OH)_2}$

(c) $2[(HCOO)_2Cu \cdot 3Cu(OH)_2] + (HCOO)_2Cu \rightarrow 3[(HCOO)_2Cu \cdot 2Cu(OH)_2]$

G. Fowles, J. Chem. Soc. (London), **107**, 128 (1915) *57*

$(HCOO)_2Cu$

Δ **III-1485**

Crystallized copper formate when heated in a dry test tube, decomposes evolving oxides of carbon and a brilliant residue of metallic copper.

$$((HCOO)_2Cu \rightarrow Cu + CO + CO_2 + H_2O)$$

F. W. Clarke, Science, **1**, 67 (1883) *73*

HC_2Cu_2O

HCl **III-1486**

Boiling hydrochloric acid decomposes cuprous acetyl oxide, giving acetylene and cuprous chloride.

$$4HC_2Cu_2O + 8HCl \rightarrow 4C_2H_2 + 8CuCl + (2H_2O + O_2)$$

Bertholet, Compt. rend., **62**, 456 (1866) *29*

$KCN \cdot 2Cu_2(CN)_2$

$Cu(NO_3)_2$ **III-1487**

The double salts of cuprous cyanide react with cupric salts to give a green precipitate which is a combination of the cuprous and cupric cyanides.

$$2[KCN \cdot 2Cu_2(CN)_2] + Cu(NO_3)_2 \rightarrow \underline{Cu(CN)_2 \cdot 2Cu_2(CN)_2} + (2KNO_3) + (2Cu_2(CN)_2)$$

A. Lallemand, Compt. rend., **58**, 751 (1864) *29*

$K_2Cu(C_2O_4)_2$

III-1488

Light

The photodecomposition of potassium cuprioxalate can probably be represented by the equation given below.
Mixed salts and oxides, the result of secondary reaction, are also formed.

$$K_2Cu(C_2O_4)_2 \rightarrow K_2C_2O_4 + CuC_2O_4$$

H. Dube and N. Dhar, J. Phys. Chem., **36**, 630 (1932) *85*

$K_6Cu_2(CN)_8$

III-1489

$CuSO_4$

A 50% solution of potassium cuprous cyanide will react with a concentrated solution of copper sulfate yielding a crystalline precipitate.

$$K_6Cu_2(CN)_8 + CuSO_4 \rightarrow \underline{K_2Cu_2Cu(CN)_6} + K_2SO_4 + 2KCN$$

P. Straus, Z. anorg. Chem., **9**, 6 (1895) *28*

$K_6Cu_2(CN)_8$

III-1490

$MnSO_4$

A solution of potassium cuprous cyanide will react with a solution of manganous sulfate yielding a white precipitate.

$$K_6Cu_2(CN)_8 + 2MnSO_4 \rightarrow \underline{K_2Cu_2Mn(CN)_6} + Mn(CN)_2 + 2K_2SO_4$$

P. Straus, Z. anorg. Chem., **9**, 6 (1895) *28*

$Na_2Cu_2Mn(CN)_6$

III-1491

NH_4NO_3

A hot solution of sodium cuprous manganous cyanide will react with a saturated solution of ammonium nitrate yielding white crystals.

$$Na_2Cu_2Mn(CN)_6 + 2NH_4NO_3 \rightarrow (NH_4)_2Cu_2Mn(CN)_6 + 2NaNO_3$$

P. Straus, Z. anorg. Chem., **9**, 6 (1895) *28*

DiO

Cl_2 **III-1492**

Didymium oxide (a mixture of praseodymium and neodymium oxides) when gently heated with chlorine, forms the oxychloride.

(a) $3DiO + Cl_2 \rightarrow Di_3O_2Cl_2 + (O)$

(b) $(3PrO + Cl_2 \rightarrow Pr_3O_2Cl_2 + O)$

(c) $3NdO + Cl_2 \rightarrow Nd_3O_2 + Cl_2 + O$

Fr. Frerichs, Ber., **7**, 798 (1874) *11*

DiO

HNO_3 **III-1493**

Didymium oxide dissolves in nitric acid forming the nitrate. The salt melts at 300°C without decomposition, forming a rose red mass.

$$DiO + 2HNO_3 \rightarrow Di(NO_3)_2 + H_2O$$

Fr. Frerichs, Ber., **7**, 800 (1874) *11*

DiO

H_2SO_4 **III-1494**

Didymium oxide dissolves in sulfuric acid to form the sulfate. It contains 3 moles of water of hydration, two, which are lost at 200°C and the third at 250°C.

(a) $DiO + H_2SO_4 + 2H_2O \rightarrow DiSO_4 \cdot 3H_2O$

(b) $DiSO_4 \cdot 3H_2O \rightarrow DiSO_4 \cdot H_2O + 2H_2O$

(c) $DiSO_4 \cdot H_2O \rightarrow DiSO_4 + H_2O$

Fr. Frerichs, Ber., **7**, 799 (1874) *11*

DiO

O_2 **III-1495**

Didymium oxide when heated for six hours in a porcelain crucible in an atmosphere of oxygen, forms a sesquioxide, which when calcined in platinum crucible evolves oxygen.

(a) $4DiO + O_2 \rightarrow 2Di_2O_3$

(b) $2Di_2O_3 + \Delta + (Pt) \rightarrow 4DiO + O_2$

Fr. Frerichs, Ber., **7**, 799 (1874) *11*

$Di(OH)_4$

III-1496

$C_5H_8O_2$

3 grams of freshly precipitated didymium hydroxide is suspended in alcohol and 6 grams of acetylacetone are added. A clear solution is obtained from which, after a time, faintly reddish fine fibered crystals separate. These are didymium acetonylacetone and melt at 152°C.

$$Di(OH)_4 + 4C_5H_8O_2 \rightarrow (C_5H_7O_2)_4Di + 4H_2O$$

A. Hantzsch and M. Wolvekamp, Ann., **331**, 340 (1904) *20*

Di_2O_3

III-1497

$C_6H_4Cl \cdot SO_3H$

$C_6H_4Br \cdot SO_3H$

$C_6H_3Cl(NO_2) \cdot SO_3H(1,3,6)$

Didymium oxide is dissolved in metachlorobenzene sulfonic acid. Prismatic needles are obtained from this solution. The same type reaction occurs with metabromobenzene sulfonic acid and chloronitrobenzene sulfonic acid (1,3,6).

(a) $Di_2O_3 + 6C_6H_4Cl \cdot SO_3H + 15H_2O \rightarrow \underline{2(Di(C_6H_4Cl \cdot SO_3)_3 \cdot 9H_2O)}$

(b) $Di_2O_3 + 6C_6H_4Br \cdot SO_3H + 15H_2O \rightarrow \underline{2(Di(C_6H_4Br \cdot SO_3)_3 \cdot 9H_2O)}$

(c) $Di_2O_3 + 6C_6H_3Cl(NO_2) \cdot SO_3H + 29H_2O \rightarrow \underline{2(Di(C_6H_3Cl(NO_2) \cdot SO_3)_3 \cdot 16H_2O)}$

O. Holmberg, Z. anorg. Chem., **53**, 83 (1907) *28*

Di_2O_3

III-1498

$C_6H_4(NO_2)SO_3H$

Didymium oxide is dissolved in metanitro benzene sulfonic acid. Crystals of didymium metanitro benzene sulfonate will be obtained from this solution.

$$Di_2O_3 + 6C_6H_4(NO_2)SO_3H + 9H_2O \rightarrow 2(Di(C_6H_4(NO_2) \cdot SO_3)_3 \cdot 6H_2O)$$

O. Holmberg, Z. anorg. Chem., **53**, 83 (1907) *28*

Di_2O_3

$C_6H_4(NO_2)SO_3H$ **III-1499**

Didymium oxide is dissolved in metanitro benzene sulfonic acid. Didymium benzene sulfonate will crystalline from the solution in thin crystals.

$$Di_2O_3 + 6C_6H_4(NO_2)SO_3H + 18H_2O \rightarrow 2(Di(C_6H_5SO_3)_3 \cdot 9H_2O) + ?$$

O. Holmberg, Z. anorg. Chem., **53**, 83 (1907) *28*

Di_2O_3

$C_{10}H_7 \cdot SO_3H$ **III-1500**

$C_{10}H_6 \cdot (NO_2) \cdot SO_3H(1,5)$

$C_{10}H_6 \cdot (NO_2) \cdot SO_3H(1,6)$

Didymium oxide is dissolved in alpha-naphthalene sulfonic acid. Crystalline needles will be obtained from this solution. The same type reaction occurs with 1,5 and 1,6-nitro-naphthalene sulfonic acid.

(a) $Di_2O_3 + 6C_{10}H_7 \cdot SO_3H + 9H_2O \rightarrow 2(Di(C_{10}H_7 \cdot SO_3)_3 \cdot 6H_2O)$

(b) $Di_2O_3 + 6C_{10}H_6(NO_2) \cdot SO_3H + 9H_2O \rightarrow 2(Di(C_{10}H_6(NO_2) \cdot SO_3)_3 \cdot 6H_2O)$

(c) $Di_2O_3 + 6C_{10}H_6(NO_2) \cdot SO_3H + 15H_2O \rightarrow 2(Di(C_{10}H_6(NO_2)SO_3)_3 \cdot 9H_2O)$

O. Holmberg, Z. anorg. Chem., **53**, 83 (1907) *28*

$Di_3O_2Cl_2$

H_2O **III-1501**

Didymium oxychloride when boiled with water forms didymium chloride and hydroxide.

$$Di_3O_2Cl_2 + 2H_2O \rightarrow DiCl_2 + 2Di(OH)_2$$

Fr. Frerichs, Ber., **7**, 798 (1874) *11*

$Dy_2(C_2O_4)_3$

III-1502

Δ

Dysprosium oxide is produced when dysprosium oxalate is ignited for several hours in a muffle furnace at 800° and finally at 900°.

$$Dy_2(C_2O_4)_3 \rightarrow Dy_2O_3 + (3CO_2 + 3CO)$$

H. C. Kremers, B. S. Hopkins and E. W. Engle, J. Am. Chem. Soc., **40**, 603 (1918) *1*

Dy_2O_3

III-1503

HCl

Dysprosium chloride is formed when the oxide is treated with hydrochloric acid.

$$Dy_2O_3 + 6HCl \rightarrow 2DyCl_3 + 3H_2O$$

J. E. Egan and C. W. Balke, J. Am. Chem. Soc., **35**, 365 (1913)
Ref., Kremers, Hopkins and Engle, ibid, **40**, 604 (1918) *1*

Dy_2O_3

III-1504

HCl

Dysprosium oxide, dissolved in hydrochloric acid and suitably concentrated to a solution containing 40–45 parts anhydrous salt for 100 parts solvent, treated with $1\frac{1}{2}$ times its volume of hydrochloric acid, and allowed to stand in a sulfuric acid desiccator, forms dysprosium chloride hexahydrate.

$$Dy_2O_3 + 6HCl + 9H_2O \rightarrow 2DyCl_3 \cdot 6H_2O$$

G. Urbain and G. Jantsch, Compt. rend., **146**, 129 (1908) *38*

Dy_2O_3

III-1505

HNO_3

When dysprosium oxide is treated with the calculated amount of nitric acid to which has been added $\frac{1}{8}$ its volume of water, dysprosium nitrate pentahydrate is formed.

$$Dy_2O_3 + 6HNO_3 + 7H_2O \rightarrow 2Dy(NO_3)_3 \cdot 5H_2O$$

G. Urbain and G. Jantsch, Compt. rend., **146**, 128 (1908) *38*

Dy_2O_3

HNO_3 **III-1506**

HCl

Dysprosium oxide dissolved in dilute nitric acid forms dysprosium nitrate pentahydrate, which is efflorescent. The oxide dissolved in hydrochloric acid forms dysprosium chloride.

(a) $Dy_2O_3 + 6HNO_3 + 7H_2O \rightarrow 2[Dy(NO_3)_3 \cdot 5H_2O]$

(b) $Dy_2O_3 + 6HCl + 9H_2O \rightarrow 2[DyCl_3 \cdot 6H_2O]$

E. Horton, J. Chem. Soc. (London), **94:2**, 189 (1908) *57*

$Er(COOH)_3$

III-1507

Δ

When erbium formate is heated to red heat very voluminous erbium oxide is formed.

(a) $2Er(COOH)_3 + \text{heat} \rightarrow Er_2O_3 + (3H_2O + 6CO)$

(b) $2Er(COOH)_3 + 3O_2 \rightarrow Er_2O_3 + (3H_2O + 6CO_2)$

P. T. Cleve, K. Sven. Vet. Ak. Bih., **2**, No. 12, 7 (1874) *10*

$ErCl_3$

III-1508

$AuCl_3$

Erbium chloride and gold chloride yield large, orange-yellow tabular crystals of hydrated erbium gold chloride.

$$ErCl_3 + AuCl_3 + 9H_2O \rightarrow ErCl_3 \cdot AuCl_3 \cdot 9H_2O$$

P. T. Cleve, K. Sven. Vet. Ak. Bih., **2**, No. 12, 5 (1874) *10*

$ErCl_3$

III-1509

HF

An excess of hydrofluoric acid added to solutions of erbium chloride or erbium nitrate produces a milky, gelatinous precipitate in a dilute solution, and a somewhat translucent, amethyst red precipitate of erbium fluoride in a concentrated solution.

(a) $ErCl_3 + 3HF \rightarrow \underline{ErF_3} + (3HCl)$

(b) $Er(NO_3)_3 + 3HF \rightarrow \underline{ErF_3} + (3HNO_3)$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Bih., **1**, No. 8, 21 (1872) *10*

$ErCl_3$

III-1510

$H_2C_2O_4$

Erbium oxalate (perhaps the hydrated salt) is precipitated as a light rose-red, crystalline compound when oxalic acid is added to a warm dilute solution of erbium chloride.

$$2ErCl_3 + 3H_2C_2O_4 \rightarrow \underline{Er_2(C_2O_4)_3} + (6HCl)$$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Bih., **1**, No. 8, 47 (1872) *10*

$ErCl_3$

H_3PO_4 **III-1511**

Nearly neutral erbium chloride and orthophosphoric acid are mixed. A copious precipitate soon forms that solidifies the whole liquid. After long heating this contracts to a small volume of pretty, rose-red, non-crystalline pieces of erbium orthophosphate.

$$ErCl_3 + H_3PO_4 \rightarrow \underline{ErPO_4} + (3HCl)$$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 41 (1872) *10*

$ErCl_3$

$HgCl_2$ **III-1512**

Mixed concentrated solutions of erbium chloride and mercuric chloride yield small, rose-red, deliquescent, cubical crystals of hydrated erbium mercury chloride.

$$ErCl_3 + 5HgCl_2 + 6H_2O \rightarrow ErCl_3 \cdot (HgCl_2)_5 \cdot (H_2O)_6$$

P. T. Cleve, K. Sven. Vet. Ak. Handl. Bih., **2**, No. 12, 5 (1874) *10*

$ErCl_3$

$K_4Fe(CN)_6$ **III-1513**

Erbium chloride (concentrated neutral solution) added to a large excess of potassium ferrocyanide solution produces an immediate, white, amorphous precipitate of the hydrated double ferrocyanide of erbium and potassium.

(a) $4ErCl_3 + 3K_4Fe(CN)_6 \rightarrow Er_4(Fe(CN)_6)_3 + (12KCl)$

(b) $Er_4(Fe(CN)_6)_3 + K_4Fe(CN)_6 + xH_2O$
$\rightarrow \underline{4KErFe(CN)_6} \cdot \underline{(H_2O)x}$

P. T. Cleve and Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 23 (1872) *10*

$ErCl_3$

III-1514

$(NH_4)_2S$

Ammonium sulfide precipitates erbium hydroxide from erbium salts, hydrogen sulfide being evolved.

(a) $2ErCl_3 + 3(NH_4)_2S + 6H_2O \rightarrow \underline{2Er(OH)_3} + (6NH_4Cl) + 3H_2S$

(b) $2Er(NO_3)_3 + 3(NH_4)_2S + 6H_2O \rightarrow \underline{2Er(OH)_3} + (6NH_4NO_3) + 3H_2S$

(c) $Er_2(SO_4)_3 + 3(NH_4)_2S + 6H_2O \rightarrow \underline{2Er(OH)_3} + (3(NH_4)_2SO_4) + 3H_2S$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 16 (1872) *10*

$ErCl_3$

III-1515

$PtCl_4$

Mixed concentrated solutions of erbium chloride and platinum chloride, evaporated over sulfuric acid, yields large, orange-red, deliquescent, well-formed tablets of hydrated double chloride of erbium and platinum.

$$ErCl_3 + PtCl_4 + 11H_2O \rightarrow ErCl_3 \cdot PtCl_4 \cdot 11H_2O$$

P. T. Cleve, K. Sven. Vet. Ak. Handl. Bih., **2**, No. 12, 4 (1874) *10*

$ErCl_3 \cdot 6H_2O$

III-1516

Δ

Crystallized erbium chloride when heated first loses water of hydration and then decomposes similarly to crystallized magnesium chloride.

(a) $ErCl_3 \cdot 6(H_2O) \rightarrow ErCl_3 + 6H_2O$

(b) $2ErCl_3 + 3H_2O \rightarrow (Er_2O_3 + 6HCl)$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 20 (1872) *10*

$Er(NO_3)_3$

HIO_4 **III-1517**

Periodic acid is added to a cold, dilute, slightly acid solution of erbium nitrate till the precipitate which first forms has dissolved. From this solution there separates on standing crystalline crusts of erbium periodate.

$$Er(NO_3)_3 + 3HIO_4 \rightarrow (Er(IO_4)_3 + 3HNO_3)$$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 31 (1872) *10*

$Er(NO_3)_3$

$H_2C_2O_4$ **III-1518**

Oxalic acid solution added to a warm strongly acid solution of erbium nitrate precipitates nona-hydrated erbium oxalate as a rose-red, crystalline powder. When precipitated cold the erbium oxalate comes down as a tough mass that can be drawn into threads and then becomes crystalline.

$$2Er(NO_3)_3 + 3H_2C_2O_4 + 9H_2O \rightarrow \underline{Er_2(C_2O_4)_3 \cdot 9H_2O} + (6HNO_3)$$

P. T. Cleve, K. Sven. Vet. Ak. Handl. Öfvers, **37**, No. 7, 7 (1880) *10*

$Er(NO_3)_3$

H_2SeO_3 **III-1519**

When erbium nitrate solution is mixed with an excess of selenous acid solution and alcohol added penta-hydrated erbium acid selenite separates as round, ball-like clusters of very fine, rose-red, needle crystals.

$$2Er(NO_3)_3 + 4H_2SeO_3 + 4H_2O \rightarrow 6HNO_3 + Er_2(SeO_3)_3 \cdot SeO_2 \cdot 5H_2O$$

P. T. Cleve, K. Sven. Vet. Ak. Handl. Öfvers., **37**, No. 7, 6 (1880) *10*

$Er(NO_3)_3$

III-1520

H_2SeO_3

NH_4OH

A somewhat dilute erbium nitrate solution is treated with an excess of selenious acid solution which has been nearly neutralized with ammonium hydroxide. An immediate, amorphous precipitate is formed, probably erbium selenite.

(a) $(2NH_4OH + H_2SeO_3 \rightarrow (NH_4)_2SeO_3 + 2H_2O)$

(b) $3(NH_4)_2SeO_3 + 2Er(NO_3)_3 \rightarrow \underline{Er_2(SeO_3)_3} + (6NH_4NO_3)$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 38 (1872) *10*

$Er(NO_3)_3$

III-1521

NaOH

Sodium hydroxide solution added to erbium nitrate solution precipitates gelatinous, voluminous erbium hydroxide with a pale amethyst red color.

$$Er(NO_3)_3 + 3NaOH \rightarrow \underline{Er(OH)_3} + (3NaNO_3)$$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 15 (1872) *10*

$Er(NO_3)_3$

III-1522

Na_2CO_3

An excess of sodium carbonate produces in an erbium nitrate solution a voluminous fluocculent precipitate which dissolves slowly and yields on evaporation large, rose-red, well-formed crystals of the hydrated double carbonate of erbium and sodium.

(a) $2Er(NO_3)_3 + 3Na_2CO_3 \rightarrow \underline{(Er_2(CO_3)_3} + 6NaNO_3)$

(b) $Er_2(CO_3)_3 + 5Na_2CO_3 + 36H_2O$
$\rightarrow Er_2CO_3 \cdot (Na_2CO_3)_5 \cdot 36H_2O$

P. T. Cleve, K. Sven. Vet. Ak. Handl. Bih., **2**, No. 12, 11 (1874) *10*

$Er(NO_3)_3$

Na_2SeO_3 **III-1523**

Excess of sodium selenite precipitates from erbium nitrate solution heavy, white, microscopic masses of penta-hydrated normal erbium selenite.

$$2Er(NO_3)_3 + 3Na_2SeO_3 + 5H_2O \rightarrow \underline{Er_2(SeO_3)_3 \cdot 5H_2O} + (6NaNO_3)$$

L. F. Nilson, Nova Acta Soc. Uppsal. [3,] **9**, No. 7, 89 (1874) *10*

$Er(NO_3)_3$

Δ **III-1524**

Erbium nitrate is decomposed by heat yielding rose-red amorphous erbium oxide.

$$4Er(NO_3)_3 + \text{heat} \rightarrow 2Er_2O_3 + (12NO_2 + 3O_2)$$

P. T. Cleve., K. Sven. Vet. Ak. Handl. Öfvers., **37**, No. 7, 4 (1880) *10*

$Er(OH)_3$

CO_2 **III-1525**

Erbium hydroxide absorbs carbon dioxide readily from the air, forming erbium carbonate.

$$2Er(OH)_3 + 3CO_2 \rightarrow Er_2(CO_3)_3 + (3H_2O)$$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 15 (1872) *10*

$Er(OH)_3$

CO_2 **III-1526**

H_2O

Carbon dioxide passed into water containing suspended erbium hydroxide, and the process repeated several times at intervals of twenty four hours, finally forms rose-red lumps of erbium carbonate.

$$2Er(OH)_3 + 3CO_2 + 3H_2O \rightarrow \underline{Er_2(CO_3)_3} + (6H_2O)$$

P. T. Cleve and Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 43 (1872) *10*

$Er(OH)_3$

III-1527

HCl

Erbium hydroxide dissolves easily in hydrochloric acid, forming erbium chloride.

$$Er(OH)_3 + 3HCl \rightarrow ErCl_3 + (3H_2O)$$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 15 (1872) *10*

$Er(OH)_3$

III-1528

HNO_3

Erbium hydroxide dissolves readily in nitric acid, forming erbium nitrate.

$$Er(OH)_3 + 3HNO_3 \rightarrow Er(NO_3)_3 + (3H_2O)$$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 15 (1872) *10*

$Er(OH)_3$

III-1529

$H_2C_4H_4O_6$

Erbium hydroxide is easily soluble in tartaric acid. The product is either the normal erbium tartrate or the erbium hydrogen tartrate.

(a) $2Er(OH)_3 + 3H_2C_4H_4O_6 \rightarrow (Er_2(C_4H_4O_6)_3 + 6H_2O)$

(b) $Er(OH)_3 + 3H_2C_4H_4O_6 \rightarrow (Er(HC_4H_4O_6)_3 + 3H_2O)$

P. T. Cleve and Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 52 (1872) *10*

$Er(OH)_3$

III-1530

H_2SO_4

Erbium hydroxide dissolves easily in sulfuric acid, forming erbium sulfate.

$$2Er(OH)_3 + 3H_2SO_4 \rightarrow Er_2(SO_4)_3 + (6H_2O)$$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 15 (1872) *10*

$Er(OH)_3$

H_2SeO_4 **III-1531**

Erbium hydroxide dissolves in selenic acid, and yields erbium selenate. On evaporation at 70° to 80° rose-red tablets of the octa-hydrated salt are obtained, but at ordinary temperature well-formed crystals of the nona-hydrated salt are obtained.

(a) $2Er(OH)_3 + 3H_2SeO_4 \rightarrow Er_2(SeO_4)_3 + (6H_2O)$

(b) $Er_2(SeO_4)_3 + 8H_2O \rightarrow Er_2(SeO_4)_3 \cdot 8(H_2O)$

(c) $Er_2(SeO_4)_3 + 9H_2O \rightarrow Er_2(SeO_4)_3 \cdot 9(H_2O)$

P. T. Cleve, K. Sven. Vet. Ak. Handl. Bih., **2**, No. 12, 9 (1874) *10*

$Er(OH)_3$

NH_4Cl **III-1532**

When erbium hydroxide is boiled in a solution of ammonium chloride it dissolves slowly.

$$Er(OH)_3 + 3NH_4Cl \rightarrow (ErCl_3 + 3NH_3 + 3H_2O)$$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 15 (1872) *10*

$Er(OH)_3$

SO_2 **III-1533**

H_2O

When sulfur dioxide is passed into water containing suspended erbium hydroxide the latter dissolves. Alcohol precipitates a hydrated mass, probably an acid salt. Heating and crystallizing yields deep red, fine, long needles.

$$Er(OH)_3 + 3SO_2 + 3H_2O \rightarrow Er(HSO_3)_3 + 3H_2O$$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 37 (1872) *10*

$Er(OH)_3$

Δ **III-1534**

Erbium oxide, made by heating erbium hydroxide to a white heat, is grayish red or rose red in color and less easily soluble in other reagents than is the oxide made by heating erbium oxalate.

$$2Er(OH)_3 + \Delta \rightarrow Er_2O_3 + (3H_2O)$$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 15 (1872) *10*

$Er(SCN)_3$

III-1535

$Hg(CN)_2$

Mixed solutions of erbium thiocyanate and mercury cyanide yield on evaporation flat, pale rose-red prisms of the hydrated double salt erbium thiocyanate mercury cyanide. These lose six molecules of water of hydration over sulfuric acid and the rest at 130°.

(a) $Er(SCN)_3 + 3Hg(CN)_2 + 12H_2O \rightarrow Er(SCN)_3 \cdot (Hg(CN)_2)_3 12H_2O$

(b) $Er(SCN)_3 \cdot (Hg(CN)_2)_3 \cdot 12H_2O \rightarrow Er(SCN)_3 \cdot (Hg(CN)_2)_3 \cdot 6H_2O + 6H_2O$

P. T. Cleve, K. Sven. Vet. Ak. Handl. Bih., **2**, No. 12, 6 (1874) *10*

$Er_2(C_2O_4)_3$

III-1536

$(NH_4)_2C_2O_4$

Erbium oxalate is soluble in boiling concentrated solution of ammonium oxalate, with the formation of the double oxalate of erbium and ammonium.

$$xEr_2(C_2O_4)_3 + y(NH_4)_2C_2O_4 \rightarrow ((Er_2(C_2O_4)_3)x \cdot ((NH_4)_2C_2O_4)y)$$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 50 (1872) *10*

$Er_2(C_2O_4)_3$

III-1537

Δ

When erbium oxalate is ignited to a red heat erbium oxide is formed as a fine powder of yellowish color with a tinge of red. A similar oxide is formed when the basic nitrate of erbium is ignited.

$$2Er_2(C_2O_4)_3 + \Delta + 3O_2 \rightarrow 2Er_2O_3 + (12CO_2)$$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 15 (1872) *10*

Er_2O_3

III-1538

$C_2H_4O_2$

The oxides of the yttrium group earths can be dissolved in acetic acid. The acetates afford a rapid method for separating erbium from the mixture.

$$(Er_2O_3 + 6C_2H_4O_2 \rightarrow 2Er(C_2H_3O_2)_3 + 3H_2O)$$

J. Allen Harris and Alan H. Cameron, Trans. Roy. Soc. Can. [III], **26**, 61 (1932) *69*

Er_2O_3

HBr **III-1539**

Erbium oxide dissolves in concentrated hydrobromic acid. When the solution is evaporated, first over a steam bath and then over potash and sulfuric acid, there separate pale rose red, deliquescent, prismatic needles of the bromide containing nine molecules of water of hydration.

(a) $Er_2O_3 + 6HBr \rightarrow 2ErBr_3 + (3H_2O)$

(b) $ErBr_3 + 9H_2O \rightarrow ErBr_3 \cdot 9H_2O$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 20 (1872) *10*

Er_2O_3

HCOOH **III-1540**

Erbium oxide dissolves in formic acid, the solution yielding rose-red, wart-like aggregates of tightly packed needle crystals of hydrated erbium formate. The water of hydration is lost at 100°.

(a) $Er_2O_3 + 6HCOOH \rightarrow 2Er(COOH)_3 + (3H_2O)$

(b) $Er(COOH)_3 + 2H_2O \rightarrow Er(COOH)_3 \cdot 2H_2O$

(c) $Er(COOH)_3 \cdot 2H_2O \rightarrow Er(COOH)_3 + 2H_2O$

P. T. Cleve, K. Sven. Vet. Ak. Handl. Bih., **2**, No. 12, 7 (1874) *10*

Er_2O_3

HCOOH **III-1541**

Erbium oxide dissolves in warm formic acid and yields di-hydrated erbium formate as a heavy, rose-red, crystalline powder.

$$Er_2O_3 + 6HCOOH + H_2O \rightarrow 2Er(CHO_2)_3 \cdot 2H_2O$$

P. T. Cleve, K. Sven. Vet. Ak. Handl. Öfvers., **37**, No. 7, 7 (1880) *10*

Er_2O_3

$H(CH_3)_2PO_4$ **III-1542**

Pale colored needles of erbium dimethyl phosphate are formed when erbium oxide is dissolved in dimethyl phosphoric acid.

$$Er_2O_3 + 6H(CH_3)_2PO_4 \rightarrow (Er_2[(CH_3)_2PO_4]_6) + (3H_2O)$$

J. C. Morgan and C. James, J. Am. Chem. Soc., **36**, 13 (1914) *1*

Er_2O_3

III-1543

$HC_2H_3O_2$

Erbium oxide, when in fine powder form, is fairly easily soluble in acetic acid, forming erbium acetate.

$$Er_2O_3 + 6HC_2H_3O_2 \rightarrow 2Er(C_2H_3O_2)_3 + (3H_2O)$$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 15 (1872) *10*

Er_2O_3

III-1544

HCl

Erbium oxide is easily soluble in hydrochloric acid with formation of erbium chloride.

$$Er_2O_3 + 6HCl \rightarrow 2ErCl_3 + (3H_2O)$$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 15 (1872) *10*

Er_2O_3

III-1545

HI

Erbium oxide dissolves in an excess of concentrated hydriodic acid. Evaporation of the solution over a water bath yields unstable crystalline masses of erbium iodide.

$$Er_2O_3 + 6HI \rightarrow 2ErI_3 + (3H_2O)$$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 21 (1872) *10*

Er_2O_3

III-1546

HNO_3

Erbium oxide is easily soluble in nitric acid, forming erbium nitrate.

$$Er_2O_3 + 6HNO_3 \rightarrow 2Er(NO_3)_3 + (3H_2O)$$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 15 (1872) *10*

Er_2O_3

III-1547

HNO_3

Erbium oxide dissolves in nitric acid yielding on evaporation pretty, red, stable crystals of erbium nitrate, probably penta-hydrated.

$$Er_2O_3 + 6HNO_3 + 7H_2O \rightarrow 2Er(NO_3)_3 \cdot 5H_2O$$

P. T. Cleve, K. Sven. Vet. Ak. Handl. Öfvers., **37**, No. 7, 4–5 (1880) *10*

Er_2O_3

HSCN **III-1548**

Erbium oxide dissolves in thiocyanic acid. The solution on evaporation over the water bath deposits radiated masses of four-sided prisms of the hexa-hydrated thiocyanate of erbium.

(a) $Er_2O_3 + 6HSCN \rightarrow 2Er(SCN)_3 + 3H_2O$

(b) $Er(SCN)_3 + 6H_2O \rightarrow Er(SCN)_3 \cdot 6H_2O$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 24 (1872) *10*

Er_2O_3

H_2SO_4 **III-1549**

Erbium oxide is fairly easily soluble in sulfuric acid, forming erbium sulfate.

$$Er_2O_3 + 3H_2SO_4 \rightarrow Er_2(SO_4)_3 + (3H_2O)$$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 15 (1872) *10*

Er_2O_3

H_2SO_4 **III-1550**

Erbium oxide dissolves in sulfuric acid yielding on evaporation pretty, rose-red, well-formed crystals of octa-hydrated erbium sulfate.

$$Er_2O_3 + 3H_2SO_4 + 5H_2O \rightarrow Er_2(SO_4)_3 \cdot 8H_2O$$

P. T. Cleve, K. Sven. Vet. Ak. Handl. Öfvers., **37**, No. 7, 4–5 (1880) *10*

Er_2O_3

H_2WO_4 **III-1551**

NaCl

Erbium oxide, tungstic acid and sodium chloride fused together yield rose-red, octahedral crystals of the 2:3 double tungstate of erbium and sodium.

$$2Er_2O_3 + 9H_2WO_4 + 6NaCl \rightarrow 6H_2O + 6HCl + (Er_2(WO_4)_3)_2 \cdot (Na_2WO_4)_3$$

A. G. Högbom, K. Sven. Vet. Ak. Handl. Öfvers., **41**, No. 5, 121 (1884) *10*

Er_2O_3

III-1552

$H_4P_2O_7$

Erbium oxide dissolves in pyrophosphoric acid. After twenty four hours very small prismatic crystals of erbium pyrophosphate separate from the liquid.

$$3H_4P_2O_7 + 2Er_2O_3 \rightarrow Er_4(P_2O_7)_3 + (6H_2O)$$

P. T. Cleve and Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 41 (1872) *10*

Er_2O_3

III-1553

$KHSO_4$

Fused potassium hydrogen sulfate dissolves erbium oxide, even though the latter has been ignited till it has become nearly insoluble in acid, forming the double sulfate of erbium and potassium.

(a) $(2KHSO_4 \rightarrow K_2SO_4 + H_2SO_4)$

(b) $(3H_2SO_4 + Er_2O_3 \rightarrow Er_2(SO_4)_3 + (3H_2O))$

(c) $(Er_2(SO_4)_3 + K_2SO_4 \rightarrow Er_2(SO_4)_3 \cdot K_2SO_4)$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 35 (1872) *10*

Er_2O_3

III-1554

NH_4Cl

When erbium oxide is boiled in a solution of ammonium chloride, the oxide goes into solution and ammonia is evolved.

$$Er_2O_3 + 6NH_4Cl \rightarrow 2ErCl_3 + 6NH_3 + (3H_2O)$$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 15 (1872) *10*

$Er_2(SO_4)_3$

III-1555

$Ba(BrO_3)_2$

Double displacement of equivalent amounts of erbium sulfate and barium bromate, separation of the precipitated barium sulfate, and evaporation of the solution over a water bath and then over sulfuric acid, forms radiated crystalline masses which fall apart into asbestos-like, long, fine prisms of erbium bromate containing nine molecules of water of hydration.

(a) $Er_2(SO_4)_3 + 3Ba(BrO_3)_2 \rightarrow \underline{3BaSO_4} + 2Er(BrO_3)_3$

(b) $Er(BrO_3)_3 + 9H_2O \rightarrow Er(BrO_3)_3 \cdot 9H_2O$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 29 (1872) *10*

$Er_2(SO_4)_3$

$Ba(C_2H_5 \cdot SO_4)_2$ **III-1556**

Solutions of erbium sulfate and barium ethyl sulfate are mixed, the barium sulfate filtered off, and the filtrate yields beautiful, fairly large, well-formed, rose-red crystals of hydrated erbium ethyl sulfate.

$$Er_2(SO_4)_3 + 3Ba(C_2H_5SO_4)_2 + 18H_2O \rightarrow \underline{3BaSO_4} + Er_2(C_2H_5)_6(SO_4)_6 \cdot 18H_2O$$

J. E. Alèn, K. Sven. Vet. Ak. Handl. Öfvers., **37**, No. 8, 41 (1880) *10*

$Er_2(SO_4)_3$

$Ba(ClO_3)_2$ **III-1557**

Solutions containing equivalent weights of barium chlorate and erbium sulfate are mixed, the precipitated barium sulfate filtered off, and the solution evaporated under reduced pressure. Deliquescent, (hydrated) erbium chlorate crystals are obtained.

$$Er_2(SO_4)_3 + 3Ba(ClO_3)_2 \rightarrow \underline{3BaSO_4} + 2Er(ClO_3)_3$$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 27 (1872) *10*

$Er_2(SO_4)_3$

$Ba(ClO_4)_2$ **III-1558**

Solutions of erbium sulfate and barium perchlorate, containing equivalent amounts of each, are mixed, the precipitated barium sulfate filtered off, and the solution evaporated over a water bath. Transparent, glasslike, crystals of (hydrated) erbium perchlorate are formed.

$$Er_2(SO_4)_3 + 3Ba(ClO_4)_2 \rightarrow \underline{3BaSO_4} + 2Er(ClO_4)_3$$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 28 (1872) *10*

$Er_2(SO_4)_3$

III-1559

$BaPt(CN)_4$

When solutions of erbium sulfate and barium platinum cyanide are mixed barium sulfate precipitates. The rose-red solution forms dark red crystals which reflect violet-blue on some faces and green on other faces, and which consists of hydrated erbium platinum cyanide.

$$3BaPt(CN)_4 + Er_2(SO_4)_3 + 21H_2O \rightarrow \underline{3BaSO_4}\downarrow + Er_2(Pt(CN)_4)_3 \cdot 21H_2O$$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 25 (1872) *10*

$Er_2(SO_4)_3$

III-1560

Δ

Erbium sulfate begins to decompose at 630° and is completely converted into the oxide at 1055°.

(a) $Er_2(SO_4)_3 \rightarrow Er_2O_3 \cdot SO_3 + 2SO_3$

(b) $Er_2(SO_4)_3 \rightarrow Er_2O_2 \cdot SO_4 + 2SO_3$

(c) $Er_2(SO_4)_3 \rightarrow Er_2O_3 + 3SO_3$

E. Wedekind and P. Hausknecht, Ber. **54** B, 253 (1921) *44*

$Er_2(SO_4)_3$

HIO_3 **III-1561**

An excess of warm, dilute solution of iodic acid added to a warm, dilute solution of erbium sulfate produces a heavy, nearly white precipitate of erbium iodate. It somewhat resembles silver chloride and contains three molecules of water of hydration. It is nearly insoluble in water.

(a) $Er_2(SO_4)_3 + 6HIO_3 \rightarrow \underline{2Er(IO_3)_3} + (3H_2SO_4)$

(b) $Er(IO_3)_3 + 3H_2O \rightarrow Er(IO_3)_3 \cdot 3H_2O$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 29 (1872) *10*

$Er_2(SO_4)_3$

K_2SO_4 **III-1562**

Solutions of erbium sulfate and potassium sulfate, mixed and evaporated over sulfuric acid, yield rose-red, crystalline crusts of tetrahydrated erbium potassium sulfate.

$$Er_2(SO_4)_3 + K_2SO_4 + 4H_2O \rightarrow Er_2(SO_4)_3 \cdot K_2SO_4 \cdot 4H_2O$$

P. T. Cleve, K. Sven. Vet. Ak. Handl. Öfvers., **37**, No. 7, 6 (1880) *10*

$Er_2(SO_4)_3$

K_2SO_4 **III-1563**

When solutions of equivalent weights of erbium sulfate and of potassium sulfate are mixed and allowed to stand for several days over gentle heat hard crusts are formed containing the double sulfate of erbium and potassium.

$$Er_2(SO_4)_3 + K_2SO_4 \rightarrow Er_2(SO_4)_3 \cdot K_2SO_4$$

P. T. Cleve and O. M. Höglund, K. Sven. Vet. Ak. Handl. Bih., **1**, No. 8, 35 (1872) *10*

$Er_2(SO_4)_3$

$MgCl_2$ **III-1564**

Erbium oxychloride is the product obtained when erbium sulfate is fused with anhydrous magnesium chloride.

$$Er_2(SO_4)_3 + MgCl_2 \rightarrow 2ErOCl + (MgSO_4 + 2SO_3)$$

K. A. Hofmann, Ber., **47**, 238 (1914)
Ref., J. L. Howe, J. Am. Chem. Soc., **37**, 541 (1915) *1*

$Er_2(SO_4)_3$

$(NH_4)_2SO_4$ **III-1565**

A concentrated mixed solution of erbium sulfate and ammonium sulfate yields small, rose-red, flat, prismatic crystals of the hydrated double sulfate of erbium and ammonium.

$$Er_2(SO_4)_3 + (NH_4)_2SO_4 + 8H_2O \rightarrow Er_2(SO_4)_3 \cdot (NH_4)_2SO_4 \cdot 8H_2O$$

P. T. Cleve, K. Sven. Vet. Ak. Handl. Bih., **2**, No. 12, 10 (1874) *10*

$Er_2(SO_4)_3$

Na_2SO_4 **III-1566**

Spontaneous evaporation of mixed solutions of erbium sulfate and sodium sulfate yields rose-red crystalline masses of the hydrated double sulfate of erbium and sodium.

$$Er_2(SO_4)_3 + 5Na_2SO_4 + 7H_2O \rightarrow Er_2(SO_4)_3 \cdot (Na_2SO_4)_5 \cdot 7H_2O$$

P. T. Cleve, K. Sven. Vet. Ak. Handl. Bih., **2**, No. 12, 11 (1874) *10*

$Er_2(SO_4)_3$

Na_2SeO_3 **III-1567**

Sodium selenite precipitates from erbium sulfate solution nonahydrated normal erbium selenite as a white to rose-colored amorphous powder.

$$Er_2(SO_4)_3 + 3Na_2SeO_3 + 9H_2O \rightarrow \underline{Er_2(SeO_3)_3 \cdot 9H_2O} + (3Na_2SO_4)$$

L. F. Nilson, Nova Acta Soc. Uppsal. [3], **9**, No. 7, 90 (1874) *10*

$Er_2(SO_4)_3$

$Pb(CH_3 \cdot SO_4)_2$ **III-1568**

Solutions of erbium sulfate and lead methyl sulfate are mixed, the lead sulfate filtered off, and the filtrate yields long, red, fragile, very deliquescent needles of hydrated erbium methyl sulfate.

$$Er_2(SO_4)_3 + 3Pb(CH_3SO_4)_2 + 18H_2O \rightarrow \underline{3PbSO_4} + Er_2(CH_3)_6(SO_4)_6 \cdot 18H_2O$$

J. E. Alèn, K. Sven. Vet. Ak. Handl. Öfvers., **37**, No. 8, 18 (1880) *10*

$Er_2(SeO_3)_3$

SeO_2 **III-1569**

H_2O

When normal erbium selenite is digested with selenium dioxide in water at 60° the 1:1:3 hydrated erbium hydrogen selenite forms as an insoluble, microscopic crystalline powder.

$$Er_2(SeO_3)_3 + SeO_2 + 4H_2O \rightarrow Er_2(SeO_3)_3 \cdot H_2SeO_3 \cdot 3H_2O$$

L. F. Nilson, Nova Acta Soc. Uppsal. [3], **9**, No. 7, 91 (1874) *10*

$Er_2(SeO_4)_3$

K_2SeO_4 **III-1570**

Evaporation of a concentrated mixed solution of erbium selenate and potassium selenate yields wart-like clusters of small, rose-red crystals of the hydrated double selenate of erbium and potassium.

$$Er_2(SeO_4)_3 + K_2SeO_4 + 8H_2O \rightarrow Er_2(SeO_4)_3 \cdot K_2SeO_4 \cdot 8H_2O$$

P. T. Cleve, K. Sven. Vet. Ak. Handl. Bih., **2**, No. 12, 9 (1874) *10*

$Er_2(SeO_4)_3$

$(NH_4)_2SeO_4$ **III-1571**

Mixed solutions of erbium selenate and ammonium selenate yield aggregates of small needle crystals of the hydrated double selenate of erbium and ammonium.

$$Er_2(SeO_4)_3 + (NH_4)_2SeO_4 + 4H_2O \rightarrow Er_2(SeO_4)_3 \cdot (NH_4)_2SeO_4 \cdot 4H_2O$$

P. T. Cleve, K. Sven. Vet. Ak. Handl. Bih., **2**, No. 12, 10 (1874) *10*

$EuCl_2$

$PbCl_2$ **III-1572**

Europous chloride in hydrochloric acid solution precipitates a black colloidal suspension of lead from lead chloride.

$$2EuCl_2 + PbCl_2 \rightarrow 2EuCl_3 + Pb$$

H. N. McCoy, J. Am. Chem. Soc., **58**, 2280 (1936) *16*

$EuCl_3$

H_2SO_4 **III-1573**

Divalent europium sulfate is formed by electrolytic reduction of europium chloride in the presence of sulfuric acid.

$$2EuCl_3 + 3H_2SO_4 \rightarrow 2EuSO_4 + (6HCl + SO_2 + O_2)$$

L. F. Yntema, J. Am. Chem. Soc., **52**, 2782 (1930)
Hughes and Pearce, J. Am. Chem. Soc., **55**, 3277 (1933) *1*

$EuCl_3$

Zn **III-1574**

In the Jones reductor trivalent europium as the chloride is reduced to the divalent form.

$$2EuCl_3 + Zn \rightarrow 2EuCl_2 + ZnCl_2$$

H. N. McCoy, J. Am. Chem. Soc., **58**, 1577 (1936) *16*

$Eu(NO_3)_3$

H_2SO_4 **III-1575**

A solution of europium nitrate, treated with a slight excess of pure sulfuric acid and then added to a large amount of 95° alc., is completely transformed into the sulfate octahydrate, a silver-white, fine crystalline needle precipitate. It becomes anhydrous when heated to 300°.

$$2Eu(NO_3)_3 + 3H_2SO_4 + 8H_2O \rightarrow (6HNO_3) + \underline{Eu_2(SO_4)_3 \cdot 8H_2O}$$

G. Jantsch, Compt. rend., **146**, 474 (1908) *38*

$Eu(OH)_3$

HCOOH **III-1576**

Europium formate is formed when europium hydroxide is dissolved in dilute formic acid.

$$2Eu(OH)_3 + 6HCOOH \rightarrow Eu_2(HCOO)_6 + 6H_2O$$

C. James and J. E. Robinson, J. Am. Chem. Soc., **35**, 758 (1913) *1*

Eu_2O_3

HCl **III-1577**

Europium trioxide in a quartz reaction flask dissolves in redistilled hydrochloric acid and conductivity water. A mixture of nitrogen and hydrogen chloride is passed in from a purifying train keeping flask temperature at 95°. A crystalline salt is formed.

$$(Eu_2O_3 + 6HCl \rightarrow 2EuCl_3 + 3H_2O)$$

E. L. Meyers and B. S. Hopkins, J. Am. Chem. Soc., **57**, 241 (1935) *3*

Eu_2O_3

HNO_3 **III-1578**

The nitrates afford a rapid method for the concentration of europium, terbium and gadolinium from an yttrium-group mixture.

$$(Eu_2O_3 + 6HNO_3 \rightarrow 2Eu(NO_3)_3 + 3H_2O)$$

J. Allen Harris and D. E. Wyllie, Trans. Roy. Soc. (Canada) [III], **26**, 63 (1932) *15*

Eu_2O_3

H_2SO_4 **III-1579**

The trivalent europium sulfate is produced when europium oxide is dissolved in dilute sulfuric acid and evaporated to dryness on a steam bath.

$$Eu_2O_3 + 3H_2SO_4 \rightarrow Eu_2(SO_4)_3 + (3H_2O)$$

G. Hughes and D. W. Pearce, J. Am. Chem. Soc, **55**, 3277 (1933) *1*

AgF

CHI_3 **III-1580**

Silver fluoride reacts at slightly elevated temperatures with iodoform in the presence of H_2O. Silver iodide and hydrogen fluoride are formed and carbon monoxide is released.

$$3AgF + CHI_3 + H_2O \rightarrow CO + 3HF + 3AgI$$

Oechsner de Coninck, Bull. soc. chim. (France) [4], **5**, 62 (1909) *31*

AgF

KOH **III-1581**

At a light red heat, silver fluoride is decomposed by potassium hydroxide.

$$(2AgF + 2KOH \rightarrow 2KF + Ag_2O + H_2O)$$

Prat., Compt. rend., **65**, 345 (1867) *29*

Ag_2F_2

CHI_3 **III-1582**

Iodoform will react with silver fluoride to produce a gas, fluoroform.

$$2CHI_3 + 3Ag_2F_2 \rightarrow 2CHF_3 + 3Ag_2I_2$$

V. Auger, Bull. soc. chim. [4], **5**, 6 (1909) *31*

AsF_3

WCl_6 **III-1583**

Arsenic trifluoride reacts with tungsten hexachloride.

$$2AsF_3 + WCl_6 \rightarrow 2AsCl_3 + WF_6$$

Mitscherlich, Pogg. Ann., **59**, 94 (1843) *25*

$BaSiF_6$

NaOH **III-1584**

A dilute solution of sodium hydroxide will dissolve a precipitate of barium fluosilicate as indicated.

$$BaSiF_6 + 4NaOH \rightarrow BaF_2 + 4NaF + Si(OH)_4$$

R. H. Carter, Anal. Ed., **3**, 146 (1931) *44*

CH_3F

ε **III-1585**

If methyl fluoride is subjected to an electric difference of potential sufficient to produce sparks, it decomposes yielding hydrogen, carbon and hydrogen fluoride.

$$4CH_3F \rightarrow 2H_2 + 4C + 2H_2F_2$$

J. N. Collie, J. Chem. Soc. (London), **85:2**, 1318 (1904) *103*

CH_3F

ε **III-1586**

When methyl fluoride is sparked above mercury at ordinary pressures, carbon, hydrogen and hydrogen fluoride are formed.

$$4CH_3F \rightarrow 4C + 4H_2 + 2H_2F_2$$

J. N. Collie, J. Chem. Soc. (London), **85: 2** 1318 (1904) *102*

$C_6H_5NH_2HF$

HNO_2 **III-1587**

A good method of introducing fluorine in the benzene ring is to treat aniline hydrofluoride with nitrous acid, and then heat the diazonium compound formed.

(a) $$C_6H_5NH_2HF + HNO_2 \rightarrow C_6H_5N_2F + 2H_2O$$

(b) $$C_6H_5N_2F + \Delta \rightarrow C_6H_5F + \dot{N}_2$$

Reed and Finger, Chem. Ind., **39**, 579 (1936) *84*

F'

III-1588

SiO_2

H_2O

F' and SiF_6'' are determined with an accuracy of 1% by acidifying the solution with a known excess of HCl and titrating with KOH and K_2SiO_3, using suitable indicators. The method is based on the reaction

$$6F^- + SiO_2 + 2H_2O \rightleftharpoons SiF_6^= + 4OH^-$$

W. Geffcken and H. Hamann, Z. anal. Chem., **114**, 51–21 (1938)
Ref., Cer. Abst., **18**, 115 (1939) *16*

F_2

III-1589

Au

Fluorine acts on gold.

$$[3F_2 + 2Au \rightarrow 2AuF_3]$$

Prat, Compt. rend., **65**, 346 (1867) *29*

F_2

III-1590

B

Fluorine unites with boron.

$$(3F_2 + 2B \rightarrow 2BF_3)$$

Prat, Compt. rend., **65**, 346 (1867) *29*

F_2

III-1591

Br^-

Fluorine removes bromine from its compounds.

$$(F_2 + 2Br^- \rightarrow 2F^- + Br_2)$$

Prat, Compt. rend., **65**, 346 (1867) *29*

F_2

III-1592

Br_2

Bromine trifluoride is formed by passing fluorine over bromine.

$$3F_2 + Br_2 \rightarrow 2BrF_3$$

G. L. Clark, J. Am. Chem. Soc., **41**, 1478 (1919) *1*

F_2

Br_2 **III-1593**

Fluorine is passed into liquid bromine contained in a flask cooled in solid carbon dioxide, yielding bromine trifluoride.

$$3F_2 + Br_2 \rightarrow 2BrF_3$$

P. Lebeau, Ann. chim. phys. [VIII], **9**, 251–52 (1906) *100*

F_2

C **III-1594**

Gaseous fluorine reacts with finely divided carbon at room temperature to give a mixture of carbon tetrafluoride and ethylene tetrafluoride.

(a) $$2F_2 + C \rightarrow CF_4$$

(b) $$2F_2 + 2C \rightarrow C_2F_4$$

B. Humiston, J. Phys. Chem., **23**, 573 (1919) *85*

F_2

$CH_3)_2CO$ **III-1595**

Mixed acetone and fluorine vapor ignite spontaneously—explosively if acetone is present in excess.

$$(CH_3)_2CO + 6F_2 \rightarrow CF_4 + C_2F_4 + 2H_2F_2 + (H_2O)$$

B. Humiston, J. Phys. Chem., **23**, 574 (1919) *85*

F_2

$COCl_2$ **III-1596**

In the vapor state at 200°C the chlorine in phosgene can be substituted by fluorine.

$$COCl_2 + F_2 \rightarrow COF_2 + Cl_2$$

B. Humiston, J. Phys. Chem., **23**, 573 (1919) *85*

F_2

III-1597

C_2Cl_4

Fluorine both substitutes and decomposes tetrachloroethylene at room temperature.

(a) $$C_2Cl_4 + 2F_2 \rightarrow C_2F_4 + 2Cl_2$$

(b) $$C_2Cl_4 + Cl_2 \rightarrow C_2Cl_6$$

(c) $$C_2F_4 \rightarrow CF_4 + C$$

B. Humiston, J. Phys. Chem., **23**, 577 (1919) *85*

F_2

III-1598

Co^{++}

Fluorine is an oxidizing agent. Cobaltous salts are oxidized to cobaltic salts.

$$2Co^{++} + F_2 \rightarrow 2Co^{+++} + 2F^-.$$

F. Fichter, Z. Elektrochem., **33**, 514 (1927) *86*

F_2

III-1599

HCl

Fluorine decomposes hydrogen chloride.

$$(F_2 + 2HCl \rightarrow 2HF + Cl_2)$$

Prat, Compt. rend., **65**, 346 (1867) *29*

F_2

III-1600

HNO_3

An explosive gas forms when fluorine reacts with moderately dilute nitric acid.

$$F_2 + 2HNO_3 \rightarrow 2NO_3F + (H_2)$$

G. H. Cady, J. Am. Chem. Soc., **56**, 2636 (1934) *1*

F_2

H_2O **III-1601**

Fluorine decomposes water at once at ordinary temperature.

(a) $(2F_2 + 2H_2O \rightarrow 4HF + O_2)$

(b) $(3F_2 + 3H_2O \rightarrow 6HF + O_3)$

Prat, Compt. rend., **65**, 346 (1867) *29*

F_2

H_2O **III-1602**

If water is dropped into gaseous fluorine the whole of the vapor assumes a deep blue color due to the formation of ozone, which is blue when concentrated.

$$3F_2 + 3H_2O \rightarrow 3H_2F_2 + O_3$$

F. M. Perkin, Trans. Faraday Soc., **4**, 81 (1908) *85*

F_2

H_2O **III-1603**

NaOH

Oxygen fluoride is produced by the reaction of fluorine with cold water or with a cold solid hydroxide of sodium, potassium, or calcium.

(a) $(3F_2 + 3H_2O \rightarrow OF_2 + H_2O_2 + 4HF)$

(b) $(2F_2 + 2NaOH \rightarrow OF_2 + 2NaF + H_2O)$

G. H. Cady, J. Am. Chem. Soc., **57**, 246 (1935) *3*

F_2

Hg **III-1604**

Fluorine was absorbed by bubbling through mercury, forming mercuric fluoride.

$$Hg + F_2 \rightarrow HgF_2$$

C. Cuthbertson and E. B. R. Prideau, Trans. Roy. Soc. (London), **205A**, 322 (1906) *105*

F_2

III-1605

I^-

Fluorine eliminates iodine from its compounds.

$$(F_2 + 2I^- \rightarrow 2F^- + I_2)$$

Prat, Compt. rend., **65**, 346 (1867) *29*

F_2

III-1606

I_2

Iodine pentafluoride is formed when fluorine is passed over iodine.

$$5F_2 + I_2 \rightarrow 2IF_5$$

G. L. Clark, J. Am. Chem. Soc., **41**, 1478 (1919) *1*

F_2

III-1607

NO

Nitric oxide was mixed with an excess of fluorine in a porcelain tube and cooled to −80°C. Nitrogen dioxyfluoride and nitrogen were formed.

$$4NO + F_2 \rightarrow 2NO_2F + N_2$$

H. Moissan and P. Lebeau, Ann. chim. phys. [VIII], **9**, 229 (1906) *100*

F_2

III-1608

NaOH

Fluorine was absorbed by bubbling it through sodium hydroxide solution, forming sodium fluoride.

$$2F_2 + 4NaOH \rightarrow 4NaF + 2H_2O + O_2$$

C. Cuthbertson and E. B. R. Prideau, Trans. Roy. Soc. (London), **205A**, 322 (1906) *105*

F_2

III-1609

Pb

Fluorine was absorbed by passing it over lead filings, forming lead fluoride.

$$Pb + F_2 \rightarrow PbF_2$$

C. Cuthbertson and E. B. R. Prideau, Trans. Roy. Soc. (London), **205A**, 322 (1906) *105*

F_2

Pt **III-1610**

Fluorine reacts with platinum.

$$(F_2 + Pt \rightarrow PtF_2)$$

Prat, Compt. rend., **65**, 346 (1867) *29*

F_2

Se **III-1611**

Fluorine at room temperature reacts only partially with selenium metal.

(a) $$2F_2 + Se \rightarrow SeF_4$$

(b) $$3F_2 + Se \rightarrow SeF_6$$

B. Humiston, J. Phys. Chem., **23**, 573 (1919) *85*

F_2

Si **III-1612**

Fluorine unites with silicon and with other metals.

$$(2F_2 + Si \rightarrow SiF_4)$$

Prat, Compt. rend., **65**, 346 (1867) *29*

F_2

ε **III-1613**

The chemical action of fluorine on aqueous solutions is the electrolytic anode reaction plus secondary effects due to the hydrofluoric acid formed. Persulfates are formed from acid sulfate solutions, perphosphates from phosphate, etc.

(a) $$2KHSO_4 + F_2 \rightarrow K_2S_2O_8 + 2HF$$

(b) $$Na_3PO_4 + H_2O + F_2 \rightarrow 2HF + Na_3PO_5$$

(c) $$2KHCO_3 + F_2 \rightarrow 2HF + K_2C_2O_6$$

(d) $$2CoSO_4 + H_2SO_4 + F_2 \rightarrow Co_2(SO_4)_3 + 2HF$$

(e) $$2MnSO_4 + H_2SO_4 + F_2 \rightarrow Mn_2(SO_4)_3 + 2HF$$

(f) $$Mn_2(SO_4)_3 + 8H_2O + 4F_2 \rightarrow 2HMnO_4 + 3H_2SO_4 + 8HF$$

(g) $$H_2CrO_4 + H_2O + F_2 \rightarrow 2HF + H_2CrO_5$$

(h) $$4H_2CrO_5 + 12HF \rightarrow 4CrF_3 + 10H_2O + 5O_2$$

N. C. Jones, J. Phys. Chem., **33**, 820 (1929) *85*

HF

III-1614

$(COOK)_2$

3 grams of neutral potassium oxalate and 2 grams of potassium fluoride are dissolved in warm 40% hydrofluoric acid. The solution is allowed to stand over phosphorus pentoxide. Colorless crystals of a double compound separate.

A similar reaction occurs when this reagent is treated with rubidium oxalate or ammonium oxalate.

$$(COOK)_2 + 2HF \rightarrow HC_2O_4K \cdot HF + KF$$

R. Weinland and W. Stille, Ann., **328**, 151 (1903) *20*

HF

III-1615

HOCl

By pouring a dilute solution of hydrogen fluoride into a solution of hypochlorous acid, fluorine chloride is obtained as a gas having a color more intense than that of chlorine.

$$(HF + HOCl \rightarrow FCl + H_2O)$$

Prat, Compt. rend., **65**, 346 (1867) *29*

HF·KF

III-1616

ε

H_2O

When an aqueous solution of H_2F_2 or a fluoride is electrolyzed, it is reasonable to expect ozone, H_2O_2, or hypofluorite as the products of the action of the nascent fluorine on water. The first of these has been detected, but only in small amounts; while the other two were not observed.

$$3F_2 + 3H_2O \rightarrow 3H_2F_2 + O_3$$

E. B. Prideau, Trans. Faraday Soc., **2**, 34 (1906) *85*

HSO_3F

CH_3COOH **III-1617**

C_2H_5COOH

A mixture of glacial acetic acid and fluosulfonic acid is distilled from a platinum retort. The fractional distillation of the distillate at 20·5° yields pure acetyl fluoride. The same type reaction occurs with propionic acid.

(a) $HSO_3F + CH_3COOH \rightarrow CH_3COF + H_2SO_4$

(b) $HSO_3F + C_2H_5COOH \rightarrow C_2H_5COF + H_2SO_4$

Wilhelm Traube and Anna Krahmer, Ber., **52**:**2**, 1293 (1919) *28*

HSO_3F

$ClCH_2COOH$ **III-1618**

$Cl_2CHCOOH$

Monochloroacetic acid will react with fluosulfonic acid yielding monochloroacetyl fluoride. The same type reaction occurs with dichloroacetic acid.

(a) $HSO_3F + ClCH_2COOH \rightarrow ClCH_2COF + H_2SO_4$

(b) $HSO_3F + Cl_2CHCOOH \rightarrow Cl_2CHCOF + H_2SO_4$

Wilhelm Traube and Anna Krahmer, Ber., **52**:**2**, 1293 (1919) *28*

H_2F_2

B_2O_3 **III-1619**

Boric oxide is weighed into copper vessel. Anhydrous hydrogen fluoride is passed in until an increase in weight is produced corresponding to slightly less than theoretical requirement.

The absorption is very rapid throughout and heat is liberated. Boron fluoride is formed but remains in solution as long as the reaction vessel is cool. The liquid is decanted from the excess boric oxide and distilled.

$$4B_2O_3 + 9H_2F_2 \rightarrow 6(HBO_2 \cdot H_2F_2) + 2BF_3$$

F. J. Sowa, J. W. Kroeger, and J. A. Nieuwland, J. Am. Chem. Soc., **57**, 454 (1935) *3*

H_2F_2

III-1620

HBO_2

Anhydrous hydrogen fluoride is passed into a previously heated equimolecular mixture of orthoboric acid and boric oxide. Dihydroxyfluoboric acid is obtained on subsequent distillation.

$$HBO_2 + H_2F_2 \rightarrow (HBO_2 \cdot H_2F_2)$$

F. J. Sowa, J. W. Kroeger, and J. A. Nieuwland, J. Am. Chem. Soc., **57**, 454 (1935) *3*

H_2F_2

III-1621

NaOH

A sample of the mixed acids, hydrofluoric and fluosilicic, is first treated with potassium nitrate, then titrated ice cold with sodium hydroxide using phenolphthalein as indicator.

(a) $$H_2F_2 + 2NaOH \rightarrow 2NaF + 2H_2O$$

(b) $$H_2SiF_6 + 2KNO_3 + 2NaOH \rightarrow K_2SiF_6 + 2NaNO_3 + 2H_2O$$

Brinton, Sarber and Stoppel, Ind. Eng. Chem., **15**, 1080 (1923) *23*

H_2F_2

III-1622

SO_3

Liquid hydrogen fluoride will react with liquid sulfur trioxide yielding a colorless liquid fluosulfonic acid.

$$H_2F_2 + 2SO_3 + 2SO_3HF$$

T. E. Thorpe and W. Kirman, Z. anorg. Chem., **3**, 63 (1893) *28*

H_2F_2

III-1623

SiO_2

When hydrofluoric acid acts on silicon dioxide, silicon tetrafluoride and water are formed.

$$2H_2F_2 + SiO_2 \rightarrow SiF_4 + 2H_2O$$

J. N. Collie, J. Chem. Soc. (London), **85**, 1318 (1904) *102*

H_2F_2

ε III-1624

If methyl fluoride is sparked by an electric arc for about five minutes in the presence of glass the hydrofluoric acid formed attacks the glass and converts the silicon dioxide to silicon fluoride which is changed to free silicon and hydrofluoric acid by the free hydrogen present.

(a) $2H_2F_2 + SiO_2 \rightarrow SiF_4 + 2H_2O$

(b) $SiF_4 + 2H_2 \rightarrow Si + 2H_2F_2$

J. N. Collie, J. Chem. Soc. (London), **85**:2, 1318 (1904) *108*

H_2SiF_6

$CaCl_2$ III-1625

NaOH

Orthosilicic acid is produced when fluosilicic acid is titrated with sodium hydroxide in presence of an excess of neutral calcium chloride solution.

$$H_2SiF_6 + 3CaCl_2 + 6NaOH \rightarrow 3CaF_2 + 6NaCl + H_4SiO_4 + 2H_2O$$

Schucht and Möller, Ber., **39**, 3693 (1906)
Ref., J. Am. Chem. Soc. **30**, 439 (1908) *1*

H_2SiF_6

KCl III-1626

NaOH

Fluosilicic acid may be determined quantitatively by adding ice and an excess of potassium chloride, then titrating with sodium hydroxide.

(a) $H_2SiF_6 + 2KCl \rightarrow K_2SiF_6 + 2HCl$

(b) $HCl + NaOH \rightarrow NaCl + H_2O$

Katherine K. Worthington and Malcolm M. Haring, Anal. Ed., **3**, 7 (1931) *44*

H_2SiF_6

NaOH III-1627

Neutralization of fluosilicic acid proceeds as follows.

$$H_2SiF_6 + 6NaOH \rightarrow 6NaF + \underline{SiO_2} + 4H_2O$$

Katherine K. Worthington and Malcolm M. Haring, Anal. Ed., **3**, 7 (1931) *44*

H_2SiF_6

III-1628

NaOH

A mixture of hydrofluoric and hydrofluosilicic acids may be determined by adding potassium nitrate and first titrating ice cold with sodium hydroxide, using phenolphthalein as indicator, and then the titrated solution heated to about 80°C and again titrated to pink end point.

(a) $$H_2F_2 + 2NaOH \rightarrow 2NaF + 2H_2O$$

(b) $$H_2SiF_6 + KNO_3 + 2NaOH \rightarrow K_2SiF_6 + 2NaNO_3 + 2H_2O$$

(c) $$K_2SiF_6 + 4NaOH \rightarrow 4NaF + K_2F_2 + Si(OH)_4$$

Brinton, Sarver and Stoppel, Ind. Eng. Chem., **15**, 1080 (1923) *1*

H_2SiF_6

III-1629

NaOH

Sodium hydroxide converts fluosilicic acid quantitatively into sodium fluoride.

$$H_2SiF_6 + 6NaOH \rightarrow 6NaF + H_4SiO_4 + 2H_2O$$

W. C. Schumb and E. L. Gamble, J. Am. Chem. Soc., **54**, 589 (1932) *16*

KF

III-1630

$AlCl_3$

HCl

When a neutral solution of potassium fluoride is added to an aqueous solution containing aluminum chloride and hydrochloric acid, the aluminum chloride is decomposed into two stable compounds, neutral to phenolphthalein.

$$AlCl_3 + 6KF + xHCl \rightarrow AlF_3 \cdot 3KF + 3KCl + xHCl$$

Walter Green and S. R. Baker, Anal. Ed., **17**, 351 (1945) *44*

KF

$Al(OH)_3$ **III-1631**

Aluminum hydroxide, formed by the neutralization of a solution of an aluminum salt to the phenolphthalein end point, reacts with potassium fluoride to liberate three moles of potassium hydroxide per mole of aluminum. The reaction is practically instantaneous in the presence of sodium potassium tartrate.

(a) $$Al^{+++} + 3OH^- \rightarrow Al(OH)_3$$

(b) $$Al(OH)_3 + 6KF \rightarrow AlF_3 \cdot 3KF + 3KOH$$

L. J. Snyder, Anal. Ed., **17**, 37 (1945) *44*

KF

KNO_3 **III-1632**

MnO_2

By heating one part of potassium fluoride with five parts of potassium nitrate, or with two parts of manganese dioxide, oxygen and fluorine are given off. It is necessary to use a platinum retort. The oxygen can be removed by passing the gas through heated barium oxide.

(a) $$(2KF + 2KNO_3 \rightarrow F_2 + O_2 + 2K_2O + 2NO)$$

(b) $$(4KF + 4MnO_2 \rightarrow 2F_2 + O_2 + 2K_2O + 4MnO)$$

Prat., Compt. rend., **65**, 345, 511 (1867) *29*

$(KF)_3HF \cdot PbF_4$

H_2O **III-1633**

Tripotassium lead hydrogen fluoride is quantitatively hydrolyzed by water.

$$2[(KF)_3\ HF \cdot PbF_4] + 4H_2O \rightarrow 3K_2F_2 + 5H_2F_2 + 2PbO_2$$

G. L. Clark, J. Am. Chem. Soc., **41**, 1484 (1919) *1*

$(KF)_3HF \cdot PbF_4$

Δ **III-1634**

Free fluorine is prepared when tripotassium lead hydrogen octafluoride is decomposed by heat above 2500°.

$$2[(KF)_3HF \cdot PbF_4] \rightarrow 3F_2 + H_2F_2 + (2K_2PbF_3 + K_2F_2)$$

B. Brauner, J. Chem. Soc., (London), **65**, 393 (1894) *1*

KHF_2

III-1635

ε

Fluorine was produced by Moissan's method. Potassium fluoride is dissolved in liquid hydrogen fluoride and electrolyzed, producing fluorine gas at the anode, and hydrogen at the cathode.

(a) $$2KF \rightarrow 2K + F_2$$

(b) $$2K + 2HF \rightarrow 2KF + H_2$$

C. Cuthbertson and E. B. R. Prideau, Trans. Roy. Soc. (London), **205A**, 322 (1906) *105*

KHF_2

III-1636

ε

C

Potassium hydrogen fluoride was electrolyzed to form fluorine. The fluorine was then passed over carbon in the form of Norit to prepare carbon tetrafluoride.

(a) $$2KHF_2 \rightarrow (2HF) + (2K) + F_2$$

(b) $$2K + 2HF \rightarrow (2KF) + (H_2)$$

(c) $$C + 2F_2 \rightarrow CF_4$$

W. Cawood and H. S. Patterson, Trans. Roy. Soc. (London), **236A**, 77–102 (1937) *105*

KHF_2

III-1637

Δ

Hydrofluoric acid is formed when potassium hydrogen fluoride is heated in a platinum vessel.

$$2KHF_2 \rightarrow H_2F_2 + (K_2F_2)$$

Fremy, Ann. Phys. [3], **47**, 5 (1856)
N. H. Furman, J. Am. Chem. Soc., **40**, 907 (1918) *1*

KSO_3F

$C_6H_5CH_2 \cdot CH_2 \cdot COONa$ **III-1638**

$C_6H_5CH_2 \cdot COONa$

The dry mixture of potassium fluosulfonate and sodium hydrocinnamate is distilled quickly from a copper retort. Impure hydrocinnamyl fluoride, a liquid, is formed which can be purified by distilling in vacuum. The same type reaction occurs with sodium phenyl acetate.

(a) $KSO_3F + C_6H_5CH_2 \cdot CH_2 \cdot COONa \rightarrow C_6H_5CH_2 \cdot CH_2 \cdot COF + KNaSO_4$

(b) $KSO_3F + C_6H_5CH_2 \cdot COONa \rightarrow C_6H_5CH_2 \cdot COF + KNaSO_4$

Wilhelm Traube and Anna Krahmer, Ber., **52:2**, 1293 (1919) *28*

K_2SiF_6

KOH **III-1639**

Potassium fluosilicate reacts quantitatively with aqueous potassium hydroxide as shown by the following equation:

$$K_2SiF_6 + 4KOH \rightarrow 6KF + SiO_2 + 2H_2O$$

Travers, Compt. rend., **173**, 714 (1921)
Ref., C. M. Smith, E. H. Hamilton, and J. J. T. Graham, J.A.O.A.C., **14**, 253 (1931) *18*

K_2SiF_6

NaOH **III-1640**

A solution of potassium fluosilicate heated to about 80°C and titrated with sodium hydroxide, using phenolphthalein as indicator will give sodium fluoride, potassium fluoride and orthosilicic acid.

$$K_2SiF_6 + 4NaOH \rightarrow 4NaF + 2KF + H_4SiO_4$$

Brinton, Sarver and Stoppel, Ind. and Eng. Chem., **15**, 1080 (1923) *23*

MF

III-1641

ICl_3

Fluorine polyhalides of cesium, rubidium, potassium and ammonium may be prepared by mixing in the cold a saturated aqueous solution of the alkali fluoride with a saturated solution of iodine trichloride. Brilliant yellow crystals separate, which are further purified by recrystallization. The salts are fairly stable and are best stored over crystals of iodine trichloride.

$$MF + ICl_3 \rightarrow MFICl_3$$

H. S. Booth, C. F. Swinehart and W. C. Morris, J. Phys. Chem., **36**, 2788 (1932) *85*

NO_2F

III-1642

H_2O

Nitrogen dioxyfluoride added to water forms nitric acid and hydrofluoric acid.

$$2NO_2F + 2H_2O \rightarrow 2HNO_3 + H_2F_2$$

H. Moissan and P. Lebeau, Ann. chim. phys., [VIII], **9**, 232 (1906) *100*

NO_3F

III-1643

H_2O

Nitrogen trioxyfluoride reacts with water to produce oxygen.

$$2NO_3F + 4H_2O \rightarrow 5O_2 + (H_2F_2 + 2NH_3)]$$

G. H. Cady, J. Am. Chem. Soc., **56**, 2636 (1934) *1*

NO_3F

III-1644

KI

Iodine is liberated when nitrogen trioxyfluoride reacts with potassium iodide.

$$2NO_3F + 2KI \rightarrow 2KNO_3 + (I_2 + F_2)$$

G. H. Cady, J. Am. Chem. Soc., **56**, 2637 (1934) *1*

NO_3F

KI **III-1645**

The reaction of fluorine with moderately dilute nitric acid produces a gaseous compound NO_3F which reacts as follows with solutions of potassium iodide.

$$NO_3F + 3KI \rightarrow KI_3 + KF + KNO_3$$

G. H. Cady, J. Am. Chem. Soc., **56**, 2637 (1934) *16*

NO_3F

KOH **III-1646**

The reaction of fluorine with moderately dilute nitric acid produces a gaseous compound, NO_3F, which reacts with solutions of potassium hydroxide as follows:

$$NO_3F + 2KOH \rightarrow \tfrac{1}{2}O_2 + KF + KNO_3 + H_2O$$

G. H. Cady, J. Am. Chem. Soc., **56**, 2637 (1934) *16*

NaF

HCl **III-1647**

Sodium fluoride is converted into the chloride by heating in dry gaseous hydrochloric acid.

$$2NaF + 2HCl \rightarrow 2NaCl + (H_2F_2)$$

Baxter, Moore, and Boylston, Proc. Am. Acad., **47**, 585
Ref., F. W. Clarke, J. Chem. Soc. (London), **101**, 1830 (1912) *57*

NaF

MgOH **III-1648**

By the action of magnesium hydroxide on sodium fluoride, sodium hydroxide and a double fluoride of magnesium and sodium are formed.

$$5NaF + 2Mg(OH)_2 \rightarrow 4NaOH + NaF\cdot 2MgF_2 \text{ (''MgOHO'') (''NaOHO'')}$$

Ch. Tissier, Compt. rend., **56**, 849 (1863) *29*

NaF

III-1649

$PbCl_2$

In determining fluorine the following reaction was used.

$$NaF + PbCl_2 \rightarrow PbFCl + NaCl$$

I. Tananaev. Z. anal. Chem., **99**, 18–21 (1934)
Ref., Cer. Abst., **14**, 174 (1935) *16*

NaF

III-1650

SiF_4

A method for the determination of fluorine is based on the decomposition of many fluorides by concentrated sulfuric acid and silica, the fluorine liberated as SiF_4 is absorbed in NaF.

$$2NaF + SiF_4 \rightarrow Na_2SiF_6$$

Shinkai, J. Soc. Chem. Ind. (Japan), **39**, 162B (1936)
Ref., Cer. Abst., **16**, 44 (1937) *16*

NaF

III-1651

SiO_2

HCl

Silica is converted directly into a fluosilicate by treatment with a soluble fluoride and acid in aqueous solution.

$$6NaF + SiO_2 + 4HCl \rightarrow Na_2SiF_6 + 4NaCl + 2H_2O$$

Travers, Compt. rend., **173**, 714 (1921)
Ref., C. M. Smith, E. H. Hamilton, and J. J. T. Graham, J.A.O.A.C., **14**, 253 (1931) *18*

$NaHF_2$

III-1652

NaOH

Sodium bifluoride is neutralized by sodium hydroxide.

$$NaHF_2 + NaOH \rightarrow Na_2F_2 + H_2O$$

Dinwiddie, Am. J. Sci. [(4)], **42**, 421 (1916)
H. Flisik, Ind. Eng. Chem., **17**, 307 (1925)
Ref., L. Hart., Anal. Ed., **1**, 135 (1929) *33*

$NaHF_2$

Δ **III-1653**

N_2

Pure sodium fluoride is formed when sodium hydrogen fluoride is heated in nitrogen or air.

$$2NaHF_2 \rightarrow Na_2F_2 + (H_2F_2)$$

D. J. McAdam, Jr. and E. F. Smith, J. Am. Chem. Soc., **34**, 593 (1912) *1*

Na_2SiF_6

Na_2CO_3 **III-1654**

Variable results and foliage injury attending the use of commercial sodium fluosilicate as an insecticide is due to the presence of varying quantities of sodium fluoride or sodium carbonate which would produce sodium fluoride, a compound injurious to delicate foliage.

$$Na_2SiF_6 + 2Na_2CO_3 + H_2O \rightarrow 6NaF + H_2SiO_3 + 2CO_2$$

R. C. Roark, Science, **63**, 431 (1926) *44*

Na_2SiF_6

KCl **III-1655**

Sodium fluosilicate is converted by potassium chloride in aqueous solution into the less soluble potassium fluosilicate.

$$Na_2SiF_6 + 2KCl \rightarrow \underline{K_2SiF_6} + 2NaCl$$

Travers, Compt. rend., **173**, 714 (1921)
Ref., C. M. Smith, E. H. Hamilton, and J. J. T. Graham, J.A.O.A.C., **14**, 253 (1931) *18*

SOClF

Hg **III-1656**

When thionyl chlorofluoride is treated with mercury at room temperature, two reactions take place, (a) being faster than (b).

(a) $$2SOClF + (Hg) \rightarrow SOCl_2 + SOF_2$$

(b) $$2SOCl_2 + 3Hg \rightarrow 2HgCl_2 + HgS + SO_2$$

H. S. Booth and F. C. Mericola, J. Am. Chem. Soc., **62**, 640 (1940) *3*

SO_3HF

III-1657

Δ

Sulfuryl difluoride is formed by distilling fluosulfonic acid.

$$2SO_3HF \rightarrow SO_2F_2 + H_2SO_4$$

T. E. Thorpe and W. Kirman, Z. anorg. Chem., **3**, 63 (1893) *28*

SbF_3

III-1658

SO_2Cl_2

$SbCl_5$

Inorganic chlorofluorides are prepared from the heating of a non-polar chloride with antimony trifluoride in the presence of an antimony pentahalide as a catalyst.

$$SbF_3 + 3SO_2Cl_2 + (SbCl_5) \rightarrow SbCl_3 + 3SO_2ClF + (SbCl_5)$$

H. S. Booth and C. V. Herrmann, J. Am. Chem. Soc., **58**, 63 (1936) *3*

TeF_6

III-1659

H_2O

If the gas tellurium hexafluoride is confined over water for a period of time (10 to 12 hours) it forms telluric acid and hydrofluoric acid. The telluric acid breaks down into tellurium trioxide and water.

(a) $$TeF_6 + 4H_2O \rightarrow H_2TeO_4 + 6HF$$

(b) $$H_2TeO_4 \rightarrow TeO_3 + H_2O$$

E. B. R. Prideaux, J. Chem. Soc. (London), **89**, :1 322 (1906) *103*

$ZnSiF_6$

III-1660

NaOH

H_2O

The fluosilicates of amphoteric substances react with alkaline solutions as follows.

(a) $$ZnSiF_6 + 4NaOH \rightarrow ZnF_2 + 4NaF + 2H_2O + \underline{SiO_2}$$

(b) $$ZnF_2 + 2H_2O \rightarrow \underline{Zn(OH)_2} + 2HF$$

(c) $$HF + NaOH \rightarrow NaF + H_2O$$

(d) $$Zn(OH)_2 + 2NaOH \rightarrow Na_2ZnO_2 + 2H_2O$$

Katherine K. Worthington and Malcolm M. Haring, Anal. Ed., **3**, 7 (1931) *44*

$Gd(BrO_3)_3$

$(H_2C_2O_4)$ **III-1661**

Gadolinium bromate is converted into the oxalate by oxalic acid.

$$2Gd(BrO_3)_3 + 3H_2C_2O_4 \rightarrow Gd_2(C_2O_4)_3 + 6HBrO_3$$

D. W. Bissell and C. James, J. Am. Chem. Soc., **38**, 873 (1916) *1*

$GdCl_3$

$AuCl_3$ **III-1662**

Solutions of gadolinium chloride and gold chloride yield on evaporation yellow, well-formed crystals of deca-hydrated gadolinium gold chloride.

$$GdCl_3 + AuCl_3 + 10H_2O \rightarrow GdCl_3 \cdot AuCl_3 \cdot 10H_2O$$

C. Benedicks, K. Sv. Vet. Ak. Handl. Öfvers., **56**, No. 7, 725 (1899) *10*

$GdCl_3$

$H_2C_2O_4$ **III-1663**

Deca-hydrated gadolinium oxalate forms a crystalline precipitate, probably best from a solution of oxalic acid and gadolinium salt solutions.

$$2GdCl_3 + 3H_2C_2O_4 + 10H_2O \rightarrow \underline{Gd_2(C_2O_4)_3 \cdot 10H_2O} + (6HCl)$$

C. Benedicks, K. Sv. Vet. Ak. Handl. Öfvers., **56**, No. 7, 732 (1899) *10*

$GdCl_3$

$PtCl_4$ **III-1664**

Gadolinium chloride and an excess of platinum tetrachloride yield on evaporation fairly stable, yellow, needle crystals of the deca-hydrated double chloride of gadolinium and platinum.

$$GdCl_3 + PtCl_4 + 10H_2O \rightarrow GdCl_3 \cdot PtCl_4 \cdot 10H_2O$$

C. Benedicks, K. Sven. Vet. Ak. Handl. Öfvers., **56**, No. 7, 725 (1899) *10*

$Gd(NO_3)_3$

$C_3H_4O_4$ **III-1665**

2.5 grams of gadolinium nitrate are dissolved in water and converted to the hydroxide by the addition of ammonium hydroxide. The solution is cooled and somewhat more than the calculated amount of malonic acid added. This solution is allowed to stand a short time and then heated to boiling. Gadolinium malonate crystallizes from the solution with eight H_2O.

(a) $Gd(NO_3)_3 + 3NH_4OH \rightarrow Gd(OH)_3 + 3NH_4NO_3$

(b) $2Gd(OH)_3 + 3C_3H_4O_4 \rightarrow (C_3H_2O_4)_3Gd_2 + 6H_2O$

H. Erdmann and F. Wirth, Ann., **361**, 204 (1908) *20*

$Gd(NO_3)_3$

$K_3Co(CN)_6$ **III-1666**

Gadolinium cobalticyanide precipitates when hot solutions of gadolinium nitrate and potassium cobalticyanide are mixed.

$$2Gd(NO_3)_3 + 2K_3Co(CN)_6 + 9H_2O \rightarrow \underline{Gd_2(CoC_6N_6)_2 \cdot 9H_2O} + (6KNO_3)$$

C. James and P. S. Willard, J. Am. Chem. Soc., **38**, 1498 (1916) *1*

$Gd(NO_3)_3$

NH_4NO_3 **III-1667**

Mixed solutions of gadolinium nitrate and ammonium nitrate yield extremely deliquescent, long prisms of hydrated gadolinium ammonium nitrate.

$$xGd(NO_3)_3 + yNH_4NO_3 + zH_2O \rightarrow (Gd(NO_3)_3)_x \cdot (NH_4NO_3)_y \cdot (H_2O)_z$$

C. Benedicks, K. Sven. Vet. Ak. Handl. Öfvers., **56**, No. 7, 726 (1899) *10*

$Gd(NO_3)_3$

$NaVO_3$ **III-1668**

When solutions of gadolinium nitrate and sodium metavanadate are mixed, hydrated gadolinium orthovanadate vanadic acid anhydride is formed.

$$2Gd(NO_3)_3 + 10NaVO_3 + 28H_2O \rightarrow (GdVO_4)_2 \cdot (V_2O_5)_4 \cdot 26H_2O + (6NaNO_3 + 4NaOH)$$

C. Benedicks, K. Sven. Vet. Ak. Handl. Öfvers., **56**, No. 7, 730 (1899) *10*

$Gd(OH)_3$

CO_2 **III-1669**

When carbon dioxide is passed into warm water in which gadolinium hydroxide is suspended, fine, microscopic needles of hydrated basic gadolinium carbonate are formed.

$$Gd(OH)_3 + CO_2 \rightarrow Gd(OH)(CO_3) \cdot H_2O$$

C. Benedicks, K. Sven. Vet. Ak. Handl. Öfvers., **56**, No. 7, 732 (1899) *10*

$Gd(OH)_3$

Δ **III-1670**

When gadolinium hydroxide is heated, gadolinium oxide is left as a white powder.

$$2Gd(OH)_3 + \Delta \rightarrow Gd_2O_3 + 3H_2O$$

C. Benedicks, K. Sven. Vet. Ak. Handl. Öfvers., **56**, No. 7, 723 (1899) *10*

$Gd_2(C_2O_4)_3$

H_2SO_4 **III-1671**

Gadolinium oxalate is converted into the sulfate by concentrated sulfuric acid.

$$Gd_2(C_2O_4)_3 + 3H_2SO_4 \rightarrow Gd_2(SO_4)_3 + (3H_2C_2O_4)$$

D. W. Bissell and C. James, J. Am. Chem. Soc., **38**, 873 (1916) *1*

Gd_2O_3

$C_6H_4(NO_2)SO_3H$ **III-1672**

Gadolinium oxide is dissolved in metanitrobenzene sulfonic acid. Large crystals of gadolinium metanitrobenzene sulfonate will be obtained from this solution.

$$Gd_2O_3 + 6C_6H_4(NO_2)SO_3H + 11H_2O \rightarrow 2(Gd(C_6H_4(NO_2)SO_3)_3 \cdot 7H_2O)$$

O. Holmberg, Z. anorg. Chem., **53**, 83 (1907) *28*

Gd_2O_3

HBr **III-1673**

Gadolinium oxide dissolves in hydrobromic acid and on evaporation over sulfuric acid yields small, pointed, rhombic tablets of hexa-hydrated gadolinium bromide.

$$Gd_2O_3 + 6HBr + 9H_2O \rightarrow 2GdBr_3 \cdot 6H_2O$$

C. Benedicks, K. Sven. Vet. Ak. Handl. Öfvers., **56**, No. 7, 724 (1899) *10*

Gd_2O_3

$H(CH_3)_2PO_4$ **III-1674**

Gadolinium dimethyl phosphate is formed when gadolinium oxide is dissolved in dimethyl phosphoric acid.

$$Gd_2O_3 + 6H(CH_3)_2PO_4 \rightarrow Gd_2[(CH_3)_2PO_4]_6 + 3H_2O$$

J. C. Morgan and C. James, J. Am. Chem. Soc., **36**, 13 (1914) *1*

Gd_2O_3

$HC_2H_3O_2$ **III-1675**

Small, clear crystals of tetrahydrated gadolinium acetate are formed, probably best when gadolinium oxide is dissolved in acetic acid.

$$Gd_2O_3 + 6HC_2H_3O_2 + 5H_2O \rightarrow 2Gd(C_2H_3O_2)_3 \cdot 4H_2O$$

C. Benedicks, K. Sven. Vet. Ak. Handl. Öfvers., **56**, No. 7, 733 (1899) *10*

Gd_2O_3

HCl **III-1676**

Gadolinium oxide is easily soluble in hydrochloric acid yielding tetragonal, pyramid-ended tablets of hexa-hydrated gadolinium chloride. Crystals are large from a neutral solution and small from an acid solution.

$$Gd_2O_3 + 6HCl + 9H_2O \rightarrow 2GdCl_3 \cdot 6H_2O$$

G. Benedicks, K. Sven. Vet. Ak. Handl. Öfvers., **56**, No. 7, 723 (1899) *10*

Gd_2O_3

HNO_3 **III-1677**

Gadolinium nitrate is formed when gadolinium oxide is dissolved in 3 normal nitric acid and the excess acid removed by boiling.

$$Gd_2O_3 + 6HNO_3 \rightarrow 2Gd(NO_3)_3 + (3H_2O)$$

F. H. Spedding and G. C. Nutting, J. Am. Chem. Soc., **55**, 499 (1933) *1*

Gd_2O_3

HNO_3 **III-1678**

Gadolinium oxide dissolves easily in nitric acid yielding large crystals of the hydrated gadolinium nitrate from neutral solution and well-formed prisms of the penta-hydrated nitrate from acid solutions.

(a) $Gd_2O_3 + 6HNO_3 + 10H_2O \rightarrow (Gd(NO_3)_3)_2 \cdot 13H_2O$

(b) $Gd_2O_3 + 6HNO_3 + 7H_2O \rightarrow 2Gd(NO_3)_3 \cdot 5H_2O$

C. Benedicks, K. Sven. Vet. Ak. Handl. Öfvers., **56**, No. 7, 723, 726 (1899) *10*

Gd_2O_3

H_2SO_4 **III-1679**

Gadolinium oxide is easily soluble in sulfuric acid yielding small, lustrous crystals of octo-hydrated gadolinium sulfate.

$$Gd_2O_3 + 3H_2SO_4 + 5H_2O \rightarrow Gd_2(SO_4)_3 \cdot 8H_2O$$

C. Benedicks, K. Sven. Vet. Ak. Handl. Öfvers., **56**, No. 7, 723, 726 (1899) *10*

Gd_2O_3

H_2SeO_4 **III-1680**

Evaporation over sulfuric acid yields large, clear, efflorescent prisms of decahydrated gadolinium selenate, probably made best by dissolving gadolinium oxide in selenic acid.

$$Gd_2O_3 + 3H_2SeO_4 + 7H_2O \rightarrow Gd_2(SeO_4)_3 \cdot 10H_2O$$

C. Benedicks, K. Sven. Vet. Ak. Handl. Öfvers., **56**, No. 7, 727 (1899) *10*

$Gd_2(SO_4)_3$

$Ba(C_2H_5SO_4)_2$ **III-1681**

Solutions of gadolinium sulfate and of barium ethyl sulfate are mixed, the barium sulfate filtered off, and the filtrate yields clear, stable crystals of nona-hydrated gadolinium ethyl sulfate.

$$Gd_2(SO_4)_3 + 3Ba(C_2H_5SO_4)_2 + 18H_2O \rightarrow \underline{3BaSO_4} + 2Gd(C_2H_5SO_4)_3 \cdot 9H_2O$$

C. Benedicks, K. Sven. Vet. Ak. Handl. Öfvers., **56**, No. 7, 729 (1899) *10*

$Gd_2(SO_4)_3$

$BaPt(CN)_4$ **III-1682**

Brilliant red crystals, with a green metallic luster, of hydrated gadolinium platinum cyanide form, probably best, from solutions of gadolinium sulfate and barium platinum cyanide.

$$Gd_2(SO_4)_3 + 3BaPt(CN)_4 + 18H_2O \rightarrow \underline{3BaSO_4} + Gd_2(Pt(CN)_4)_3 \cdot 18H_2O$$

C. Benedicks, K. Sven. Vet. Ak. Handl. Öfvers., **56**, No. 7, 725 (1899) *10*

$Gd_2(SO_4)_3$

H_2S **III-1683**

Anhydrous gadolinium sulfate is heated to a dull red heat in an atmosphere of hydrogen sulfhide. Gadolinium trisulfide is formed.

$$Gd_2(SO_4)_3 + 3H_2S \rightarrow Gd_2S_3 + 3H_2SO_4$$

H. Erdmann and F. Wirth, Ann., **361**, 216 (1908) *20*

$Gd_2(SO_4)_3$

K_2SO_4 **III-1684**

Gadolinium sulfate in an excess of potassium sulfate yields on evaporation on the water bath small crystals of the di-hydrated double sulfate of gadolinium and potassium.

$$Gd_2(SO_4)_3 + K_2SO_4 + 2H_2O \rightarrow Gd_2(SO_4)_3 \cdot K_2SO_4 \cdot 2H_2O$$

C. Benedicks, K. Sven. Vet. Ak. Handl. Öfvers., **56**, No. 7, 727 (1899) *10*

$Gd_2(SO_4)_3$

Na_2SO_4 **III-1685**

Gadolinium sodium sulfate is produced when gadolinium sulfate and sodium sulfate solutions are mixed.

$$Gd_2(SO_4)_3 + Na_2SO_4 + 2H_2O \rightarrow Gd_2(SO_4)_3 \cdot Na_2SO_4 \cdot 2H_2O$$

D. W. Bissell and C. James, J. Am. Chem. Soc., **38**, 875 (1916) *1*

$Gd_2(SeO_3)_3$

H_2SeO_3 **III-1686**

Mixed solutions of gadolinium selenite and selenious acid yield clusters of microscopic needles of hexa-hydrated gadolinium hydrogen selenite.

$$Gd_2(SeO_3)_3 + H_2SeO_3 + 6H_2O \rightarrow Gd_2(SeO_3)_3 \cdot H_2SeO_3 \cdot 6H_2O$$

C. Benedicks, K. Sven. Vet. Ak. Handl. Öfvers., **56**, No. 7, 728 (1899) *10*

$Gd_2(SeO_4)_3$

K_2SeO_4 **III-1687**

Mixed solutions of gadolinium selenate and potassium selenate yield on evaporation microscopic needles of tetra-hydrated gadolinium potassium selenate.

$$Gd_2(SeO_4)_3 + 3K_2SeO_4 + 4H_2O \rightarrow Gd_2(SeO_4)_3 \cdot (K_2SeO_4)_3 \cdot 4H_2O$$

C. Benedicks, K. Sven. Vet. Ak. Handl. Öfvers., **56**, No. 7, 728 (1899) *10*

Ga

III-1688

Br_2

Gallium tribromide and triiodide may be prepared by the direct combination of the elements in the absence of air.

(a) $$2Ga + 3Br_2 \rightarrow 2GaBr_3$$

(b) $$2Ga + 3I_2 \rightarrow 2GaI_3$$

W. C. Johnson and J. B. Parsons, J. Phys. Chem., **34**, 1214 (1930) *85*

Ga

III-1689

Cl_2

Gallium trichloride is prepared by the action of chlorine diluted with nitrogen on hot gallium. The product is distilled in a stream of nitrogen to remove the excess chlorine and is further purified by sublimation *in vacuo*.

$$(2Ga + 3Cl_2 \rightarrow 2GaCl_3)$$

A. W. Laubengayer and F. B. Schirmer, J. Am. Chem. Soc., **62**, 1578 (1940) *8*

Ga

III-1690

HCl

Gallium dissolves in hydrochloric acid yielding gallium trichloride and hydrogen.

$$2Ga + 6HCl \rightarrow 2GaCl_3 + 3H_2$$

L. F. Nilson and O. Pettersson, K. Sven. Vet. Ak. Handl. Bih., **14**, II, No. 6, 13 (1888) *10*

Ga

III-1691

HCl

Gallium is soluble in concentrated HCl yielding gallium trichloride.

$$2Ga + 6HCl \rightarrow 2GaCl_3 + 3H_2$$

Hoffman, B. S. J. Res., **13**, 668 (1934) *93*

Ga

HF **III-1692**

Gallium fluoride trihydrate is prepared by treating the metal and its oxide with excess of 50% hydrofluoric acid and evaporating.

(a) $2Ga + 6HF + 6H_2O \rightarrow 2(GaF_3 \cdot 3H_2O) + 3H_2$

(b) $Ga_2O_3 + 6HF + 3H_2O \rightarrow 2(GaF_3 \cdot 3H_2O)$

W. C. Johnson and J. Parsons, J. Phys. Chem., **36**, 2592 (1932) *86*

Ga

HNO_3 **III-1693**

Gallium metal is passive to cold HNO_3, but dissolves on heating.

$$Ga + 6HNO_3 \rightarrow Ga(NO_3)_3 + 3H_2O + 3NO_2$$

Treadwell and Hall, Analytical Chemistry, 5th ed., **1**, 540 (1919) *93*

Ga

$Hg(C_2H_5)_2$ **III-1694**

Metallic mercury is formed when a mixture of gallium and mercury diethyl is heated to 165° in an atmosphere of nitrogen.

$$3Hg(C_2H_5)_2 + 2Ga \rightarrow 3Hg + 2Ga(C_2H_5)_3$$

L. M. Dennis and W. Patnode, J. Am. Chem. Soc., **54**, 184 (1932) *1*
16

Ga

NH_3 **III-1695**

Gallium nitride is prepared by reacting gallium with ammonia gas at 900°–1000°C.

$$2Ga + 2NH_3 \rightarrow 2GaN + 3H_2$$

W. C. Johnson, J. Parsons, and M. C. Crew, J. Phys. Chem., **36**, 2652, (1932) *85*

$GaBr_3$

III-1696

NH_3

Gallium tribromide and gallium triiodide react with liquid ammonia at −35.5°C to form ammoniates which are stable at room temperature in the absence of water.

(a) $GaBr_3 + 6NH_3 \rightarrow GaBr_3 \cdot 6NH_3$

(b) $GaI_3 + 6NH_3 \rightarrow GaI_3 \cdot 6NH_3$

(c) $GaBr_3 \cdot 6NH_3 + 6H_2O \rightarrow GaBr_3 \cdot 6H_2O + 6NH_3$

W. C. Johnson and J. Parsons, J. Phys. Chem., **36**, 2594 (1932) *85*

$Ga(CH_3)_2Cl$

III-1697

Na

When dimethyl gallium chloride is treated with one equivalent of sodium in liquid ammonia, dimethyl gallium is obtained (a). The free dimethylgallium combines with one or more molecules of ammonia. When the solvent ammonia is evaporated, the remaining solid decomposes on warming to room temperature (b).

(a) $(CH_3)_2GaCl + Na \rightarrow (CH_3)_2Ga + NaCl$

(b) $(CH_3)_2Ga \cdot NH_3 \rightarrow (CH_3)_2Ga \cdot NH_2 + \frac{1}{2}H_2$

C. A. Kraus and F. E. Toonder, J. Am. Chem. Soc., **55**, 3552 (1933) *16*

$Ga(CH_3)_2Cl$

III-1698

Na

NH_3

Dimethyl gallium chloride is reduced when treated with liquid ammonia solution of sodium. The dimethyl gallium formed combines with one or more molecules of ammonia and this salt decomposes on heating with evolution of hydrogen.

(a) $(CH_3)_2GaCl + Na \rightarrow (CH_3)_2Ga + NaCl$

(b) $(CH_3)_2Ga + NH_3 \rightarrow (CH_3)_2GaNH_3$

(c) $2(CH_3)_2Ga \cdot NH_3 \rightarrow 2(CH_3)_2GaNH_2 + H_2$

C. A. Kraus and F. E. Toonder, J. Am. Chem. Soc., **55**, 3552 (1933) *1*

$Ga(CH_3)_3$

B_2H_6 **III-1699**

When trimethyl gallium is treated with an excess of diborane at room temperature metallic gallium is deposited at the end of 3 hours (a). At −45° dimethylgallium borohydride is formed (b).

(a) $Ga(CH_3)_3 + 3B_2H_6 \rightarrow Ga + 3CH_3B_2H_5 + 3/2H_2$

(b) $2Ga(CH_3)_3 + 3B_2H_6 \rightarrow 2(CH_3)_2GaBH_4 + 2CH_3B_2H_5$

H. I. Schlesinger, H. C. Brown and G. W. Schaeffer, J. Am. Chem. Soc., **65**, 1787 (1943) *16*

$Ga(CH_3)_3$

Li **III-1700**

$C_2H_5NH_2$

Reaction between lithium and trimethyl gallium in ethylamine at liquid ammonia temperatures took place somewhat slowly, but at the end of twelve hours, one atomic equivalent of lithium had reacted (a), further reaction occurs as more lithium is added, (b). On distilling the ethylamine from the colorless reaction mixture hydrogen is evolved, (c).

(a) $(CH_3)_3Ga \cdot C_2H_5NH_2 + Li \rightarrow (CH_3)_3Ga \cdot C_2H_5NHLi + \frac{1}{2}H_2$

(b) $2(CH_3)_3Ga \cdot C_2H_5NH_2 + 2Li \rightarrow Li_2[(CH_3)_3Ga]_2 + 2C_2H_5NH_2$

(c) $Li_2[Ga(CH_3)_3]_2 + 2C_2H_5NH_2 \rightarrow 2LiC_2H_5NHGa(CH_3)_3 + H_2$

C. A. Kraus and F. E. Toonder, J. Am. Chem. Soc., **55**, 3550 (1933) *16*

$Ga(CH_3)_3$

Na **III-1701**

NH_3

When trimethyl gallium is treated with metallic sodium in liquid ammonia, one atom of sodium is converted to sodium amide with the evolution of hydrogen, and the amide ion forms a coordinated compound with two molecules of trimethyl gallium (a). Further addition of sodium reduces the trimethyl gallium to a negative trimethyl gallium ion (b).

(a) $2(CH_3)_3Ga \cdot NH_3 + Na \rightarrow [Ga(CH_3)_3]_2 \cdot NaNH_2 + \frac{1}{2}H_2 + NH_3$

(b) $2(CH_3)_3Ga \cdot NH_3 + 2Na \rightarrow Na_2[Ga(CH_3)_3]_2 + 2NH_3$

C. A. Kraus and F. E. Toonder, J. Am. Chem. Soc., **55**, 3549 (1933) *16*

$Ga(C_2H_5)_3$

III-1702

H_2O

Triethyl gallium hydrolyzes with cold water producing ethane and diethyl gallium hydroxide.

$$Ga(C_2H_5)_3 + H_2O \rightarrow Ga(C_2H_5)_2OH + C_2H_6$$

L. M. Dennis and W. Patnode, J. Am. Chem. Soc., **54**, 187 (1932) *1* *16*

$Ga(C_2H_5)_3$

III-1703

H_2O

HCl

Ethane is evolved when triethyl gallium is treated with water at room temperature.
Hydrochloric acid added to the product forms diethyl gallium chloride.

(a) $Ga(C_2H_5)_3 + H_2O \rightarrow Ga(C_2H_5)_2OH + C_2H_6$

(b) $Ga(C_2H_5)_2OH + HCl \rightarrow Ga(C_2H_5)_2Cl + H_2O$

L. M. Dennis and W. Patnode, J. Am. Chem. Soc., **54**, 186 (1932) *1*

$Ga(C_2H_5)_3$

NH_3

III-1704

Gallium triethyl ammine is formed when gallium triethyl and ammonia are brought together in a bulb immersed in an ice bath.

$$Ga(C_2H_5)_3 + NH_3 \rightarrow Ga(C_2H_5)_3 \cdot NH_3$$

L. M. Dennis and W. Patnode, J. Am. Chem. Soc., **54**, 186 (1932) *1*

$Ga(C_2H_5)_3 \cdot NH_3$

H_2O

III-1705

Gallium triethyl ammine (formed by the reaction of gallium triethyl with ammonia) is decomposed by water with the evolution of ammonia and ethane and the formation of diethyl gallium hydroxide.

$$Ga(C_2H_5)_3 \cdot NH_3 + H_2O \rightarrow Ga(C_2H_5)_2OH + NH_3 + C_2H_6$$

L. M. Dennis and W. Patnode, J. Am. Chem. Soc., **54**, 187 (1932) *1* *16*

$Ga(C_2H_5)_3 \cdot NH_3$

H_2SO_4 **III-1706**

Diethyl gallium sulfate is formed when gallium triethylammine is treated with sulfuric acid.

$$2Ga(C_2H_5)_3 \cdot NH_3 + H_2SO_4 \rightarrow (Ga(C_2H_5)_2)_2SO_4 + 2NH_3 + 2C_2H_6$$

L. M. Dennis and W. Patnode, J. Am. Chem. Soc., **54**, 187 (1932) *1*

$Ga(C_2H_5)_3 \cdot NH_3$

KOH **III-1707**

The potassium salt of diethyl gallium hydroxide is formed when gallium triethylammine is treated and warmed with potassium hydroxide.

(a) $Ga(C_2H_5)_3 \cdot NH_3 + KOH \rightarrow Ga(C_2H_5)_2OH + (KNH_2 + C_2H_6)$

(b) $Ga(C_2H_5)_2OH + KOH \rightarrow Ga(C_2H_5)_2OK + H_2O$

L. M. Dennis and W. Patnode, J. Am. Chem. Soc., **54**, 187 (1932) *1*

$GaCl_3$

Ga **III-1708**

Gallium dichloride is obtained from gallium trichloride by means of metallic gallium.

$$2GaCl_3 + Ga \rightarrow 3GaCl_2$$

L. F. Nilson and O. Pettersson, K. Sven. Vet. Ak. Handl. Bih., **14**, II, No. 6, 15 (1888) *10*

$GaCl_3$

$K_4Fe(CN)_6$ **III-1709**

A precipitate of gallium ferrocyanide is formed when gallium chloride is titrated electrometrically with potassium ferrocyanide in the presence of ferricyanide.

$$4GaCl_3 + 3K_4Fe(CN)_6 \rightarrow \underline{Ga_4(Fe(CN)_6)_3} + 12KCl$$

H. O. Kerschman and J. B. Ramsey, J. Am. Chem. Soc., **50**, 1634 (1928) *1*

$GaCl_3$

NH_3 **III-1710**

Gallium hydroxide precipitates when a solution of gallium chloride is diluted with water and a slight excess of ammonia added.

$$GaCl_3 + 3NH_3 + 3H_2O \rightarrow Ga(OH)_3 + 3NH_4Cl$$

H. O. Kerschman and J. B. Ramsey, J. Am. Chem. Soc., **50**, 1632 (1928) *1*

$GaCl_3$

NaOH **III-1711**

The precipitated oxide of gallium consists of the hydrous monohydrate.

$$2GaCl_3 + 6NaOH + 3H_2O \rightarrow Ga_2O_3 \cdot 3H_2O + 6NaCl$$

A. B. Weiser and W. O. Milligan, J. Phys. Chem., **42**, 677 (1938) *85*

$GaCl_3$

NaOH **III-1712**

Gallium trichloride, when dissolved in excess NaOH, forms sodium gallate the analogue of sodium orthoaluminate.

(a) $$GaCl_3 + 3NaOH \rightarrow \underline{Ga(OH)_3} + 3NaCl$$

(b) $$Ga(OH)_3 + 3NaOH \rightarrow Na_3GaO_3 + (3H_2O)$$

Hoffman, B. S. J. Res., **13**, 669 (1934) *93*

$GaCl_3$

Na_2CO_3 **III-1713**

A hot concentrated solution of sodium carbonate is added to a dilute solution of gallium chloride and the mixture held at the boiling point to give complete precipitation of the gallium oxide. The gelatinous precipitate is washed free of chloride ion with hot water. For the formation of an alpha gallium oxide this gelatinous precipitate should be heated in a muffle furnace at 425°; for the formation of the beta form ignition must be at 600°.

$$(2GaCl_3 + 3Na_2CO_3 \rightarrow Ga_2O_3 + 3CO_2 + 6NaCl)$$

A. W. Laubengayer and H. R. Engle, J. Am. Chem. Soc., **61**, 1210 (1939) *3*

$Ga(NO_3)_3$

$K_4Fe(CN)_6$ **III-1714**

When potassium ferrocyanide is added to a solution of a gallium salt, slightly acidified with HCl, and the mixture digested at 70° for 30 minutes, gallium ferrocyanide precipitates.

$$4Ga(NO_3)_3 + 3K_4Fe(CN)_6 \rightarrow \underline{Ga_4[Fe(CN)_6]_3} + 12KNO_3$$

Hillebrand and Lundell, Applied Inorganic Analysis, p. 385 (1929) *93*

$Ga(OH)_3$

H_2SeO_4 **III-1715**

Gallium selenate precipitated when gallium hydroxide is treated with an amount of selenic acid insufficient to dissolve it and digested for several hours.

$$2Ga(OH)_3 + 3H_2SeO_4 \rightarrow \underline{Ga_2(SeO_4)_3} + (6H_2O)$$

L. M. Dennis and J. A. Bridgman, J. Am. Chem. Soc., **40**, 1557 (1918) *1*

$Ga(OH)_3$

Δ **III-1716**

Gallium oxide is formed when gallium hydroxide is ignited.

$$2Ga(OH)_3 \rightarrow Ga_2O_3 + (3H_2O)$$

L. M. Dennis and J. A. Bridgman, J. Am. Chem. Soc., **40**, 1545 (1918) *1*

$Ga_2(SeO_4)_3$

Cs_2SeO_4 **III-1717**

Cesium gallium selenate alum is produced when a solution containing gallium selenate and cesium selenate and some free selenic acid is evaporated at room temperature.

$$Ga_2(SeO_4)_3 + Cs_2SeO_4 + 24H_2O \rightarrow 2CsGa(SeO_4)_2 \cdot 12H_2O$$

L. M. Dennis and J. A. Bridgman, J. Am. Chem. Soc., **40**, 1558 (1918) *1*

$Ga_2(SO_4)_3$

III-1718

$(NH_4)_2SO_4$

Gallium sulfate and ammonium sulfate in solution form a double salt of gallium ammonium sulfate (of octahedral structure)

$$Ga_2(SO_4)_3 + (NH_4)_2SO_4 + 24H_2O \rightarrow Ga_2(SO_4)_3 \cdot (NH_4)_2SO_4 \cdot 24H_2O$$

A. Henninger, Ber. (I), **9**, 64 (1876) *26*

$Ga_2(SO_4)_3$

III-1719

NaN_3

Gallium hydroxide is precipitated when solid sodium triazoate is added to a slightly acid solution of gallium sulfate and the solution boiled vigorously.

$$2Ga_2(SO_4)_3 + 12NaN_3 + 12H_2O \rightarrow \underline{4Ga(OH)_3} + (6Na_2SO_4 + 4NH_3 + 16N_2)$$

L. M. Dennis and J. A. Bridgman, J. Am. Chem. Soc., **40**, 1545 (1918) *1*

$Ga_2(SO_4)_3$

III-1720

Na_2SO_3

Gallium hydroxide precipitates when a slightly acid solution of gallium sulfate is treated with solid sodium sulfite and boiled.

$$Ga_2(SO_4)_3 + 3Na_2SO_3 + 3H_2O \rightarrow \underline{2Ga(OH)_3} + (3Na_2SO_4 + 3SO_2)$$

L. M. Dennis and J. A. Bridgman, J. Am. Chem. Soc., **40**, 1546 (1918) *1*

$Ga_2(SO_4)_3$

III-1721

ε

When an ammoniacal solution of gallium sulfate is electrolyzed, free gallium is deposited on the cathode.

$$Ga_2(SO_4)_3 \rightarrow 2Ga + 3SO_4^{=}$$

A. Henninger, Ber. **9:1** 64 (1876) *26*

$Ga_2(SO_4)_3$

Δ **III-1722**

The thermal decomposition of gallium sulfate yields the oxide:

$$Ga_2(SO_4)_3 \rightarrow Ga_2O_3 + 3SO_3$$

G. Marchal, J. Chim. phys., **22**, 559 (1925) *69*

$Na_2[(CH_3)_3Ga]_2$

NH_4Br **III-1723**

When ammonium bromide is added to a reaction mixture containing trimethyl-gallium and metallic sodium in liquid ammonia, trimethyl-gallium is regenerated from the reduced trimethyl-gallium ion with the evolution of hydrogen and formation of sodium bromide.

$$Na_2[(CH_3)_3Ga]_2 + 2NH_4Br \rightarrow 2(CH_3)_3Ga \cdot NH_3 + 2NaBr + H_2$$

C. A. Kraus and F. E. Toonder, J. Am. Chem. Soc., **55**, 3550 (1933) *16*

Na_3GaO_3

NH_4OH **III-1724**

Gallium is recovered from sodium gallate by the addition of dilute HCl until neutral, followed by precipitation with NH_4OH in slight excess.

(a) $Na_3GaO_3 + 6HCl \rightarrow GaCl_3 + [3NaCl + 3H_2O]$

(b) $GaCl_3 + 3NH_4OH \rightarrow \underline{Ga(OH)_3} + [3NH_4Cl]$

Hoffman, B. S. J. Res., **13**, 672 (1934) *93*

Na_3GaO_3

ε **III-1725**

Gallium is deposited electrolytically from a solution of a gallium salt in 10% sodium hydroxide using 2–5 amps. and 4–5 volts.

$$4Na_3GaO_3 + 6H_2O \rightarrow 4Ga + (12NaOH + 3O_2)$$

Hoffman, B. S. J. Res., **13**, 666 (1934) *93*

$C_2H_5GeI_3$

Ag_2O **III-1726**

Monoethyl germanic oxide is formed when an aqueous solution of ethyl germanium triiodide is heated with freshly prepared silver oxide.

$$2C_2H_5GeI_3 + Ag_2O + 2H_2O \rightarrow (C_2H_5GeO)_2O + (2AgI + 4HI)$$

E. A. Flood, J. Am. Chem. Soc., **55**, 4936 (1933) *1*

$C_2H_5GeI_3$

NH_3 **III-1727**

An insoluble precipitate forms when liquid ammonia is condensed with ethyl germanium triiodide.

$$C_2H_5GeI_3 + 4NH_3 \rightarrow \underline{C_2H_5GeN} + 3NH_4I$$

E. A. Flood, J. Am. Chem. Soc., **55**, 4937 (1933) *1*

C_2H_5GeN

H_2O **III-1728**

Ethyl germanium nitride hydrolyzes with the formation of the oxide.

$$2C_2H_5GeN + 3H_2O \rightarrow (C_2H_5GeO)_2O + 2NH_3$$

E. A. Flood, J. Am. Chem. Soc., **55**, 4938 (1933) *1*

$(C_2H_5)_4Ge$

Br_2 **III-1729**

A good yield of triethyl germanium bromide is formed when tetraethyl germanium in ethyl bromide solution reacts with bromine at 40°.

$$(C_2H_5)_4Ge + Br_2 \rightarrow (C_2H_5)_3GeBr + (C_2H_5Br)$$

C. A. Kraus and E. A. Flood, J. Am. Chem. Soc., **54**, 1635 (1932) *1*

$(C_6H_5)_3GeBr$

$AgNO_3$ **III-1730**

Triphenyl germanium bromide in alcoholic solution reacts with silver nitrate.

$$[(C_6H_5)_3GeBr]_2 + 2AgNO_3 \rightarrow [(C_6H_5)_3Ge]_2O + \underline{2AgBr} + (NO + NO_2 + O_2)$$

G. D. Morgan and J. D. K. Drew, J. Chem. Soc. (London), **127**, 1760 (1925) *25*

$(C_6H_5)_3GeBr$

$BrC_6H_4N(CH_3)_2$ **III-1731**

Na

One mole triphenyl germanium bromide is refluxed with two moles para-bromodimethyl aniline in xylene solution in the presence of an excess of sodium.

$$(C_6H_5)_3GeBr + BrC_6H_4N(CH_3)_2 + 2Na \rightarrow (C_6H_5)_3GeC_6H_4N(CH_3)_2 + 2NaBr$$

Orndorff, Tabern and Dennis, J. Phys. Chem., **30**, 1049 (1926) *25*

$(C_6H_5)_3GeBr$

$BrC_6H_5OCH_3Mg$ **III-1732**

Triphenyl germanium bromide is treated with anisyl magnesium bromide.

$$(C_6H_5)_3GeBr + BrMgC_6H_5OCH_3 \rightarrow (C_6H_5)_3GeC_6H_5OCH_3 + MgBr_2$$

W. R. Orndorff, D. L. Tabern and L. M. Dennis, J. Am. Chem. Soc., **49**, 2512 (1927) *1*

$(C_6H_5)_3GeBr$

$BrMgC_6H_4CH_3$ **III-1733**

An ether solution of triphenyl germanium bromide is treated with para-tolylmagnesium bromide.

$$(C_6H_5)_3GeBr + BrMgC_6H_4CH_3 \rightarrow (C_6H_5)_3GeC_6H_4CH_3 + MgBr_2$$

W. R. Orndorff, D. L. Tabern and L. M. Dennis, J. Am. Chem. Soc., **49**, 2512 (1927) *1*

$(C_6H_5)_3GeBr$

C_2H_5MgBr **III-1734**

Triphenyl germanium bromide reacts with ethyl magnesium bromide.

$$(C_6H_5)_3GeBr + C_2H_5MgBr \rightarrow \underline{(C_6H_5)_3GeC_2H_5} + MgBr_2$$

W. R. Orndorff, D. L. Tabern and L. M. Dennis, J. Am. Chem. Soc., **49**, 2512 (1927) *1*

$(C_6H_5)_3GeBr$

H_2O **III-1735**

Triphenyl germanium bromide reacts with water.

$$2(C_6H_5)_3GeBr + H_2O \rightarrow \underline{(C_6H_5)_3Ge \cdot O \cdot Ge(C_6H_5)_3} + 2HBr$$

G. D. Morgan and J. D. K. Drew, J. Chem. Soc. (London), **127**, 1760 (1925) *25*

$(C_6H_5)_3GeBr$

Na **III-1736**

Triphenyl germanium bromide is dissolved in dry xylene and heated with an excess of sodium.

$$[(C_6H_5)_3GeBr]_2 + 2Na \rightarrow (C_6H_5)_3Ge \cdot Ge(C_6H_5)_3 + 2NaBr$$

G. D. Morgan and J. D. K. Drew, J. Chem. Soc. (London), **127**, 1760 (1925) *25*

$[(C_6H_5)_3Ge]_2O$

HCl **III-1737**

Triphenyl germanium oxide dissolved in dry petrolic ether is treated with hydrogen chloride.

$$((C_6H_5)_3Ge)_2O + 2HCl \rightarrow \underline{2(C_6H_5)_3GeCl} + H_2O$$

W. R. Orndorff, D. L. Tabern and L. M. Dennis, J. Am. Chem. Soc., **49**, 2512 (1927) *1*

$(C_6H_5)_4Ge$

Br_2 **III-1738**

Tetraphenyl germanium in carbon tetrachloride solution is boiled for several hours with bromine.

$$(C_6H_5)_4Ge + Br_2 \rightarrow (C_6H_5)_3GeBr + (C_6H_5Br)$$

C. A. Kraus and Foster, J. Am. Chem. Soc., **49**, 457 (1927)
Ref., W. R. Orndorff, D. L. Tabern and L. M. Dennis, ibid., 2512 (1927) *1*

Ge

Br_2 **III-1739**

When heated gently, germanium burns in bromine vapor with the formation of germanium tetrabromide.

$$Ge + 2Br_2 \rightarrow GeBr_4$$

Winkler, J. f. prakt. Chem., **36**, 188
Ref., J. H. K., Am. Chem. J., **10**, 246 (1888) *1*

Ge

Br_2 **III-1740**

Germanium tetrabromide is formed when powdered germanium reacts with bromine at 220°.

$$Ge + 2Br_2 \rightarrow GeBr_4$$

L. M. Dennis and F. E. Hance, J. Am. Chem. Soc., **44**, 300 (1922) *1*

Ge

Cl_2 **III-1741**

Germanium reacts with chlorine in a stream of nitrogen at 80°–180°.

$$Ge + 2Cl_2 \rightarrow GeCl_4$$

Laubengayer and Tabern, J. Phys. Chem., **30**, 1047 (1926)
Winkler, J. f. prakt. Chem., **142**, 177 (1886) *7*

Ge

III-1742

Cl_2

Germanium tetrachloride is formed when powdered germanium is heated in a current of chlorine.

$$Ge + 2Cl_2 \rightarrow GeCl_4$$

L. M. Dennis and F. E. Hance, J. Am. Chem. Soc., **44**, 304 (1922) *1*

Ge

III-1743

$GeCl_4$

Metallic germanium, powdered and fused under salt to remove traces of germanium oxide, was treated with germanium tetrachloride at 430°F in vacuo. White germanium dichloride is formed.

$$Ge + GeCl_4 \rightarrow 2GeCl_2$$

L. M. Dennis and H. L. Hunter, J. Am. Chem. Soc., **51**, 1151 (1929) *16*

Ge

III-1744

$GeCl_4$

HCl

Germanium chloroform is prepared by passing the vapor of germanium tetra chloride over heated germanium forming germanium dichloride (?) and causing this product to unite with hydrogen chloride.

(a) $$Ge + GeCl_4 \rightarrow 2GeCl_2$$

(b) $$GeCl_2 + HCl \rightarrow GeHCl_3$$

L. M. Dennis, W. R. Orndorff and D. L. Tabern, J. Phys. Chem., **30**, 1049 (1926)
L. M. Dennis and H. L. Hunter, J. Am. Chem. Soc., **51**, 1151 (1929) *1*

Ge

GeS_2

III-1745

A mixture of germanium and germanium sulfide is heated in a current to CO_2.

$$Ge + GeS_2 \rightarrow 2GeS$$

Winkler, J. f. prakt. Chem., **142**, 215 (1886) *25*

Ge

HCl **III-1746**

Germanium reacts with hydrochloric acid.

(a) $$Ge + 3HCl \rightarrow GeHCl_3 + H_2$$

(b) $$Ge + 4HCl \rightarrow GeCl_4 + 2H_2$$

Winkler, J. f. prakt. Chem. [2], **35**, 391 (1887)
Ref., L. M. Dennis, W. R. Orndorff and D. L. Tabern, J. Phys. Chem., **30**, 1039 (1926)

25

Ge

HCl **III-1747**

Germanium chloroform is produced when hydrogen chloride is passed over heated germanium.

$$Ge + 3HCl \rightarrow GeHCl_3 + (H_2)$$

Winkler, J. f. prakt. Chem., **36**, 188
Ref., J. H. K., Am. Chem. J., **10**, 246 (1888)

1

Ge

HCl **III-1748**

Germanium chloroform and germanium tetrachloride are the products when hydrogen chloride is passed over heated germanium.

$$3Ge + 10HCl \rightarrow 2GeHCl_3 + GeCl_4 + (4H_2)$$

Laubengayer and Corey, J. Phys. Chem., **30**, 1043 (1926)
L. M. Dennis and H. L. Hunter, J. Am. Chem. Soc., **51**, 1151 (1929)

1

Ge

HNO_3 **III-1749**

Germanium is oxidized by nitric acid.

$$Ge + 4HNO_3 \rightarrow GeO_2 + (4NO_2 + 2H_2O)$$

L. M. Dennis, W. R. Orndorff and D. L. Tabern, J. Am. Chem. Soc., **45**, 2034 (1923)

1
10

Ge

III-1750

H_2

Germanium is heated in a stream of pure hydrogen to 750–800°.

$$Ge + 2H_2 \rightarrow GeH_4$$

J. H. Müller and N. H. Smith, J. Am. Chem. Soc., **44**, 1909 (1922) *25*

Ge

III-1751

H_2

Germanium hydride is produced when a mixture of 3 parts of germanium and 2 parts of magnesium turnings is heated to a little below red heat in a current of hydrogen.

$$Ge + 2H_2 \rightarrow GeH_4$$

L. M. Dennis, R. B. Corey and R. W. Moore, J. Am. Chem. Soc., **46**, 659 (1924) *1*

Ge

III-1752

H_2O_2

Finely powdered germanium is easily dissolved in a 3% solution of hydrogen peroxide at 100°.

$$Ge + 2H_2O_2 \rightarrow GeO_2 + 2H_2O$$

L. M. Dennis, K. Tressler and F. E. Hance, J. Am. Chem. Soc., **45**, 2046 (1923) *1*

Ge

III-1753

H_2S

Germanium and sulfur unite to form the sulfide at the dissociation temperature of hydrogen sulfide, 400–440°.

(a) $H_2S \rightarrow H_2 + S$

(b) $Ge + S \rightarrow GeS$

L. M. Dennis, K. Tressler and F. E. Hance, J. Am. Chem. Soc., **45**, 2045 (1923) *1*

Ge

H_2S **III-1754**

Germanium is heated to 350° in the presence of hydrogen sulfide.

$$Ge + H_2S \rightarrow GeS + H_2$$

L. M. Dennis, W. R. Orndorff and D. L. Tabern, J. Am. Chem. Soc., **45**, 2034 (1923) *1*

Ge

H_2SO_4 **III-1755**

Sulfuric acid acts on germanium yielding sulfur dioxide, water and a germanium sulfate which is decomposed by water into white germanium oxide and sulfuric acid.

(a) $$Ge + 4H_2SO_4 \rightarrow Ge(SO_4)_2 + 2SO_2 + 4H_2O$$

(b) $$Ge(SO_4)_2 + 2H_2O \rightarrow GeO_2 + 2H_2SO_4$$

P. T. Cleve, Sv. Kem., Tidsk., **1**, 38 (1889) *25*

Ge

H_3PO_4 **III-1756**

Germanium hydride is formed when a 50% solution of orthophosphoric acid is electrolyzed using a germanium cathode.

$$3Ge + 12H^+ + HPO_4^- + 6H_2O \rightarrow 3GeH_4 + (4H_3PO_4 + 3O_2)$$

L. M. Dennis, R. B. Corey and R. W. Moore, J. Am. Chem. Soc., **46**, 658 (1924) *1*

Ge

$HgBr_2$ **III-1757**

Germanium tetrabromide is produced when powdered germanium is heated with mercuric bromide.

$$Ge + 2HgBr_2 \rightarrow GeBr_4 + (2Hg)$$

Winkler, J. f. prakt. Chem., **36**, 188
Ref., J. H. K., Am. Chem. J., **10**, 246 (1888) *1*

Ge

$HgBr_2$ **III-1758**

A mixture of germanium and mercuric bromide is heated.

$$Ge + 4HgBr_2 \rightarrow GeBr_4 + (4HgBr)$$

Winkler, J. f. prakt. Chem. [2], **35**, 391 (1887) *25*

Ge

HgCl **III-1759**

A mixture of germanium and mercurous chloride is heated in the presence of nitrogen.

$$Ge + 4HgCl \rightarrow GeCl_4 + 4Hg$$

F. M. Brewer and L. M. Dennis, J. Phys. Chem., **31**, 1527 (1927) *25*

Ge

$HgCl_2$ **III-1760**

A mixture of germanium and mercuric chloride is heated.

$$Ge + 4HgCl_2 \rightarrow GeCl_4 + (4HgCl)$$

Winkler, J. f. prakt. Chem. [2], **35**, 391 (1887) *25*

Ge

I_2 **III-1761**

Germanium reacts with iodine vapor when heated to 250–360° in a stream of carbon dioxide.

(a) $$Ge + 2I_2 \rightarrow GeI_4$$
(b) $$Ge + I_2 \rightarrow GeI_2$$

Winkler, J. f. prakt. Chem. [2], **36**, 193 (1887)
L. M. Dennis and F. E. Hance, J. Am. Chem. Soc., **44**, 2854 (1922) *1*

Ge

I_2 **III-1762**

Germanium tetraiodide is formed when powdered germanium is heated in a current of iodine vapor. Germanous iodide forms at 440° from the tetraiodide.

(a) $$Ge + 2I_2 \rightarrow GeI_4$$

(b) $$GeI_4 \rightarrow GeI_2 + (I_2)$$

Winkler, J. f. prakt. Chem., **142**, (N. S. 34) 177 (1886) *1*

Ge

NH_3 **III-1763**

Finely divided germanium reacts with gaseous ammonia at temperatures ranging from 600–750° to produce germanic nitride.

$$3Ge + 4NH_3 \rightarrow Ge_3N_4 + 6H_2$$

G. H. Morey and W. C. Johnson, J. Am. Chem. Soc., **54**, 3603 (1932) *1*
Ref., Warren C. Johnson, J. Am. Chem. Soc., **52**, 5160 (1930) *16*

Ge

Mg **III-1764**

Powdered germanium was mixed with magnesium and heated in a stream of hydrogen, slightly below redness.

$$Ge + 2Mg \rightarrow Mg_2Ge$$

L. M. Dennis, Z. anorg. Chem., **174**, 126 (1928) *25*

Ge

Mg **III-1765**

Magnesium germanide is formed when powdered germanium is heated with an excess of magnesium filings in an atmosphere of hydrogen at 800° for 15 minutes.

$$2Mg + Ge \rightarrow Mg_2Ge$$

C. A. Kraus and E. S. Carney, J. Am. Chem. Soc., **56**, 765 (1934) *1*

Ge

III-1766

O_2

Germanium dioxide is formed when metallic germanium is heated in a current of pure oxygen at 730°.

$$Ge + O_2 \rightarrow GeO_2$$

L. M. Dennis, K. Tressler and F. E. Hance, J. Am. Chem. Soc., **45**, 2045 (1923) *1*

Ge

III-1767

O_2

Powdered germanium glows when heated in air and yields white germanium oxide, which is slightly soluble in water to a sour liquid from which small rhombohedral crystals of germanium oxide are formed on evaporation.

$$Ge + O_2 \rightarrow GeO_2$$

P. T. Cleve, Sv. Kem. Tidsk., **1**, 38 (1889) *10*

Ge

III-1768

SO_2

Germanium is heated to 350° in a stream of sulfur dioxide.

$$3Ge + 2SO_2 \rightarrow 2GeO_2 + GeS_2$$

L. M. Dennis, W. R. Orndorff and D. L. Tabern, J. Am. Chem. Soc., **45**, 2034 (1923) *1*

Ge

III-1769

SO_2

A mixture of germanium disulfide and oxide is formed when crystalline germanium is heated at 510°–530° in a current of pure sulfur dioxide.

$$3Ge + 2SO_2 \rightarrow GeS_2 + 2GeO_2$$

L. M. Dennis, K. Tressler and F. E. Hance, J. Am. Chem. Soc., **45**, 2046 (1923) *25*

Ge

Se **III-1770**

Powdered germanium and selenium in stochiometric proportion are heated to 500°C in sealed tube filled with carbon dioxide. Product is purer than by other methods.

(a) $(Ge + Se \rightarrow GeSe)$
(b) $(Ge + 2Se \rightarrow GeSe_2)$

B. N. Ivanoff-Emin, J. Gen. Chem. (USSR), **10**, 1814 (1940) *60*

$GeBr_2$

Br_2 **III-1771**

Germanous bromide reacts with bromine.

$$GeBr_2 + Br_2 \rightarrow GeBr_4$$

F. M. Brewer, J. Phys. Chem., **31**, 1817 (1927) *25*

$GeBr_2$

HBr **III-1772**

Germanium bromoform is produced by the action of gaseous HBr on $GeBr_2$.

$$GeBr_2 + HBr \rightarrow GeHBr_3$$

F. M. Brewer and L. M. Dennis, J. Phys. Chem., **31**, 1538 (1927) *85*

$GeBr_2$

Δ **III-1773**

Germanous bromide is heated.

$$2GeBr_2 + \Delta \rightarrow Ge + GeBr_4$$

F. M. Brewer and L. M. Dennis, J. Phys. Chem., **31**, 1527 (1927) *25*

$GeBr_4$

C_6H_5MgBr **III-1774**

Germanic bromide reacts with phenyl magnesium bromide in excess.

(a) $GeBr_4 + 4C_6H_5MgBr \rightarrow Ge(C_6H_5)_4 + 4MgBr_2$

(b) $GeBr_4 + 2C_6H_5MgBr \rightarrow Ge(C_6H_5)_2Br_2 + 2MgBr_2$

(c) $GeBr_4 + C_6H_5MgBr \rightarrow GeC_6H_5Br_3 + MgBr$

G. T. Morgan and H. D. K. Drew, J. Chem. Soc. (London), **127**, 1760 (1925) *25*

$GeBr_4$

H_2O **III-1775**

Germanium tetrabromide hydrolyzes producing the hydroxide.

$$GeBr_4 + 4H_2O \rightarrow \underline{Ge(OH)_4} + (4HBr)$$

L. M. Dennis and F. E. Hance, J. Am. Chem. Soc., **44**, 302 (1922) *1*

$GeBr_4$

KOH **III-1776**

Germanium tetrabromide is changed into potassium germanate by aqueous potassium hydroxide.

$$GeBr_4 + 4KOH \rightarrow K_4GeO_4 + (4HBr)$$

L. M. Dennis and F. E. Hance, J. Am. Chem. Soc., **44**, 303 (1922) *1*

$GeBr_4$

NaOH **III-1777**

Germanic bromide reacts quantitatively with a solution of sodium hydroxide.

$$GeBr_4 + 6NaOH \rightarrow Na_2GeO_3 + 4NaBr + 3H_2O$$

Baxter and Copper, J. Phys. Chem., **29**, 1372 (1925) *7*

$GeBr_4$

Zn **III-1778**

Germanic bromide reacts with freshly prepared zinc.

$$GeBr_4 + Zn \rightarrow ZnBr_2 + GeBr_2$$

F. M. Brewer and L. M. Dennis, J. Phys. Chem., **31**, 1527 (1927) *7*

$Ge(CH_3)_4$

HBr **III-1779**

Hydrogen bromide was distilled over into germanium tetramethyl in the presence of anhydrous aluminum bromide in vacuo. The two substances in the vapor phase form drops of liquid around the catalyst. Fractional distillation with liquid air of the material volatile above −100° yields trimethyl germanium bromide.

$$Ge(CH_3)_4 + HBr \rightarrow Ge(CH_3)_3Br + CH_4$$

L. M. Dennis and W. I. Patnode, J. Am. Chem. Soc., **52**, 2779 (1930) *16*

$Ge(C_2H_5)_4$

O_2 **III-1780**

Germanium tetraethyl burns in air.

$$Ge(C_2H_5)_4 + 14O_2 \rightarrow \underline{(GeO_2} + 8CO_2 + 10H_2O)$$

L. M. Dennis and F. E. Hance, J. Am. Chem. Soc., **47**, 370 (1925) *1*

$GeCl_2$

Br_2 **III-1781**

A colorless liquid consisting of germanium tetrabromide and tetrachloride is formed when germanium dichloride reacts with bromine.

$$2GeCl_2 + 2Br_2 \rightarrow GeBr_4 + GeCl_4$$

L. M. Dennis and H. L. Hunter, J. Am. Chem. Soc., **51**, 1154 (1929) *1*

$GeCl_2$

Cl_2 **III-1782**

Chlorine rapidly attacks germanium dichloride with the formation of pure germanium tetrachloride.

$$GeCl_2 + Cl_2 \rightarrow GeCl_4$$

L. M. Dennis and H. L. Hunter, J. Am. Chem. Soc., **51**, 1154 (1929) *1*

$GeCl_2$

HCl **III-1783**

When hydrogen chloride gas is passed over germanium dichloride at 30–50°C, germanium chloroform forms readily.

$$GeCl_2 + HCl \rightarrow GeHCl_3$$

L. M. Dennis, W. R. Orndorff, and D. L. Tabern, J. Phys. Chem., **30**, 1050 (1926) *85*

$GeCl_2$

H_2O **III-1784**

Germanium dichloride reacts vigorously when moist oxygen is passed over it. The hydrogen chloride liberated reacts with unchanged dichloride producing germanium chloroform.

(a) $$GeCl_2 + 2H_2O \rightarrow Ge(OH)_2 + 2HCl$$

(b) $$GeCl_2 + HCl \rightarrow GeHCl_3$$

L. M. Dennis and H. L. Hunter, J. Am. Chem. Soc., **51**, 1154 (1929) *1*
16

$GeCl_2$

H_2S **III-1785**

From a hydrochloric acid solution of germanous oxide hydrogen sulfide precipitates red-brown germanous sulfide.

$$GeCl_2 + H_2S \rightarrow \underline{GeS} + 2HCl$$

P. T. Cleve, Sv. Kem. Tidsk., **1**, 39 (1889) *10*

$GeCl_2$

H_2S **III-1786**

Hydrogen sulfide reacts on germanium dichloride at room temperature producing germanium sulfide and hydrogen chloride.

$$GeCl_2 + H_2S \rightarrow GeS + 2HCl$$

L. M. Dennis and H. L. Hunter, J. Am. Chem. Soc., **51**, 1154 (1929) *1*
16

$GeCl_2$

NaOH **III-1787**

When germanous chloride is treated with sodium hydroxide, germanous hydroxide precipitates as a yellow to brownish yellow powder. Potassium hydroxide and ammonium hydroxide act similarly.

(a) $GeCl_2 + 2NaOH \rightarrow \underline{Ge(OH)_2} + [2NaCl]$

(b) $GeCl_2 + 2KOH \rightarrow \underline{Ge(OH)_2} + [2KCl]$

(c) $GeCl_2 + 2NH_4OH \rightarrow \underline{Ge(OH)_2} + [2NH_4Cl]$

P. T. Cleve, Sv. Kem. Tidsk., **1**, 39 (1889) *10*

$GeCl_2$

O_2 **III-1788**

Dry oxygen acts slowly upon germanium dichloride. After about 15 minutes the dichloride begins to turn white and a volatile substance identified as germanium tetrachloride is formed.

$$2GeCl_2 + O_2 \rightarrow GeO_2 + GeCl_4$$

L. M. Dennis and H. L. Hunter, J. Am. Chem. Soc., **51**, 1153 (1929) *1*
16

$GeCl_4$

CH_3MgBr **III-1789**

Germanic chloride reacts with methyl magnesium bromide.

$$GeCl_4 + 4CH_3MgBr \rightarrow Ge(CH_3)_4 + (2MgBr_2 + 2MgCl_2)$$

L. M. Dennis and F. E. Hance, J. Phys. Chem., **30**, 1055 (1926) *1*

$GeCl_4$

C_2H_5MgBr **III-1790**

Germanic chloride reacts with ethyl magnesium bromide.

$$GeCl_4 + 4C_2H_5MgBr \rightarrow Ge(C_2H_5)_4 + (2MgCl_2 + 2MgBr_2)$$

D. L. Tabern, W. R. Orndorff and L. M. Dennis, J. Am. Chem. Soc., **47**, 2039 (1925) *1*

$GeCl_4$

C_2H_5OH **III-1791**

Na

Germanic chloride is treated with ethyl alcohol in the presence of sodium.

$$GeCl_4 + 4C_2H_5OH + 4Na \rightarrow Ge(OC_2H_5)_4 + (4NaCl + 2H_2)$$

D. L. Tabern, W. R. Orndorff and L. M. Dennis, J. Am. Chem. Soc., **47**, 2039 (1925) *1*

$GeCl_4$

$C_4HgMgBr$ **III-1792**

Germanic chloride reacts with n-butyl magnesium bromide in large excess.

$$GeCl_4 + 4C_4HgMgBr \rightarrow Ge(C_4Hg)_4 + (2MgBr_2 + 2MgCl_2)$$

D. L. Tabern, W. R. Orndorff and L. M. Dennis, J. Am. Chem. Soc., **49**, 2512 (1927) *1*

$GeCl_4$

$C_6H_5CH_2MgCl$ **III-1793**

Germanic chloride reacts with benzyl magnesium chloride in the presence of benzyl chloride, when dissolved in dry xylene.

$$GeCl_4 + 4C_6H_5CH_2MgCl \rightarrow Ge(CH_2C_6H_5)_4 + 4MgCl_2$$

W. R. Orndorff, D. L. Tabern and L. M. Dennis, J. Am. Chem. Soc., **49**, 2512 (1927) *1*

$GeCl_4$

$C_6H_5CH_3Br$ **III-1794**

Na

Germanic chloride reacts with p-bromotoluene in the presence of sodium.

$$GeCl_4 + 4C_6H_5CH_3Br + 8Na \rightarrow Ge(C_6H_5CH_3)_4 + (4NaBr + 4NaCl)$$

D. L. Tabern, W. R. Orndorff and L. M. Dennis, J. Am. Chem. Soc., **47**, 2039 (1925) *1*

$GeCl_4$

C_6H_5MgBr **III-1795**

Germanic chloride reacts with phenyl magnesium bromide.

$$GeCl_4 + 4C_6H_5MgBr \rightarrow Ge(C_6H_5)_4 + (2MgCl_2 + 2MgBr_2)$$

D. L. Tabern, W. R. Orndorff and L. M. Dennis, J. Am. Chem. Soc., **47**, 2039 (1925) *1*

$GeCl_4$

Ge **III-1796**

Germanic chloride vapor is passed over metallic Ge at a high temperature.

$$GeCl_4 + Ge \rightarrow 2GeCl_2$$

L. M. Dennis, Z. anorg. Chem., **174**, 102 (1928) *25*

$GeCl_4$

H **III-1797**

A solution of germanium tetrachloride will react with nascent hydrogen yielding germanium tetrahydride.

$$GeCl_4 + 8H \rightarrow GeH_4 + 4HCl$$

E. Voegelen, Z. anorg. Chem., **30**, 325 (1902) *28*

$GeCl_4$

H_2O **III-1798**

Germanium tetrachloride hydrolyzes in the presence of water or water vapor giving the oxide and HCl.

$$GeCl_4 + 2H_2O \rightleftharpoons GeO_2 + 4HCl$$

1

L. M. Dennis and E. B. Johnson, J. Am. Chem. Soc., **45**, 1386 (1923) *16*

$GeCl_4$

H_2S **III-1799**

Germanium sulfide is precipitated from a solution of germanium chloride by hydrogen sulfide.

$$GeCl_4 + 2H_2S \rightarrow \underline{GeS_2} + (4HCl)$$

J. H. Müller, J. Am. Chem. Soc., **43**, 1088 (1921) *1*

$GeCl_4$

H_2S **III-1800**

When hydrogen sulfide is passed into a solution of germanium oxide in hydrochloric acid, which yields germanium chloride, white germanic sulfide precipitates.

$$GeCl_4 + 2H_2S \rightarrow \underline{GeS_2} + (4HCl)$$

P. T. Cleve, Sv. Kem. Tidsk., **1**, 39 (1889) *10*

$GeCl_4$

H_2Se **III-1801**

Germanium diselenide is precipitated from solution of GeO_2 in 6*N* hydrochloric acid by hydrogen selenide. The precipitate is orange-yellow.

(a) $$GeO_2 + 4HCl \rightarrow GeCl_4 + 2H_2O$$

(b) $$(GeCl_4 + 2H_2Se \rightarrow \underline{GeSe_2} + 4HCl)$$

B. N. Ivanoff-Emin, J. Gen. Chem. (USSR), **10**, 1813 (1940) *60*

$GeCl_4$

NH_3 **III-1802**

Germanium tetrachloride is completely ammonolyzed in liquid ammonia to germanic imide.

$$GeCl_4 + 6NH_3 \rightarrow Ge(NH)_2 + 4NH_4Cl$$

R. Schwarz and P. W. Schenk, Ber., **63**, 296 (1930) *1*
W. C. Johnson, G. H. Morey and A. E. Kott, J. Am. Chem. Soc., **54**, 4279 (1932) *16*

$GeCl_4$

NH_3 **III-1803**

Liquid germanic chloride reacts with ammonia.

$$GeCl_4 + 6NH_3 \rightarrow \underline{GeCl_4 \cdot 6NH_3}$$

W. Pugh and J. S. Thomas, J. Chem. Soc. (London), 1051 (1926) *25*

$GeCl_4$

SbF_3 **III-1804**

$(SbCl_5)$

The fluorination of germanium tetrachloride by means of antimony trifluoride in the presence of antimony pentachloride yields monofluorotrichlorogermane, difluorodichlorogermane, trifluoromonochlorogermane, germanium tetrafluoride.

(a) $[SbF_3 + GeCl_4 \rightarrow GeCl_3F + SbClF_2$

(b) $SbF_3 + GeCl_4 \rightarrow GeCl_2F_2 + SbCl_2F$

(c) $SbF_3 + GeCl_4 \rightarrow GeClF_3 + SbCl_3$

(d) $4SbCl_2F + GeCl_4 \rightarrow GeF_4 + 4SbCl_3]$

H. S. Booth and W. C. Morris, J. Am. Chem. Soc., **58**, 90 (1936) *3*

$GeCl_4$

$Zn(C_2H_5)_2$ **III-1805**

When germanium chloride and zinc ethyl are mixed, germanium ethyl is formed as a colorless liquid with onion-like odor.

$$GeCl_4 + 2Zn(C_2H_5)_2 \rightarrow Ge(C_2H_5)_4 + 2ZnCl_2$$

P. T. Cleve, Sv. Kem. Tidsk., **1**, 39 (1889) *10*

L. M. Dennis and F. E. Hance, J. Am. Chem. Soc., **47**, 370 (1925) *16*

C. Winkler, J. f. prakt. Chem., **144**, 177 (1887) *1*

$GeCl_4$

Sodium Oleate **III-1806**

Germanium soaps may be prepared by reaction of a salt of germanium with a salt of a fatty acid. Opt. temp. is 40–70°C. Reaction of oleic salt is shown as an example.

$$4CH_3(CH_2)_7HC{:}CH(CH_2)_7COONa + GeCl_4 \rightarrow [CH_3(CH_2)_7HC{:}CH(CH_2)_7COO]_4Ge + 4NaCl$$

Brit. Pat. 395, 406, July 17, 1933
Ref., Oil and Soap, **11**, 4, 77 (1934) *111*

$GeCl_4 \cdot 6NH_3$

H_2O **III-1807**

Germanic chloride hexammoniate reacts with water.

$$GeCl_4 \cdot 6NH_3 + 4H_2O \rightarrow GeCl_4 \cdot 4NH_3 \cdot 4H_2O + 2NH_3$$

W. Pugh and J. S. Thomas, J. Chem. Soc. (London), Part 2, p. 1051, (1926) *25*

GeF_4

Ge **III-1808**

Germanic fluoride is passed over crystallized Ge at 350°.

(a) $$GeF_4 + Ge \rightarrow 2GeF_2$$

(b) $$2GeF_2 \rightarrow Ge + GeF_4 \text{ (above 350°)}$$

L. M. Dennis, Z. anorg. Chem., **174**, 115 (1928) *25*

GeF_4

H_2O **III-1809**

Hydrofluogermanic acid is produced by conducting the vapors of germanium fluoride into water.

$$2GeF_4 + 2H_2O \rightarrow H_2GeF_6 + (GeO_2 + H_2F_2)$$

Winkler, J. f. prakt. Chem., **36**, 188 (1887)
Ref., J. H. K., Am. Chem. J., **10**, 246 (1888) *1*

GeF_4

KF **III-1810**

Potassium fluoride and germanium fluoride form hexagonal crystals of the double germanium potassium fluoride.

$$GeF_4 + 2KF \rightarrow GeF_4 \cdot (KF)_2$$

P. T. Cleve, Sv. Kem. Tidsk., **1**, 39 (1889) *10*

GeF_4

KHF_2 **III-1811**

A solution of germanic fluoride is treated with potassium hydrogen fluoride.

$$GeF_4 + 2KHF_2 \rightarrow K_2GeF_6 + (H_2F_2)$$

G. Krüss and L. F. Nilson, Ber., **20**, 1696 (1887) *25*

GeH

Cl_2 **III-1812**

Dry chlorine reacts with germanium monohydride producing germanium tetra chloride. Bromine and iodine vapors act similarly.

$$2GeH + 5Cl_2 \rightarrow 2GeCl_4 + 2HCl$$

L. M. Dennis and N. A. Skow, J. Am. Chem. Soc., **52**, 2371 (1930) *1*
16

GeH

$KMnO_4$ **III-1813**

H_2SO_4

Germanium mono-hydride is quantitatively oxidized by potassium permanganate acidulated with sulfuric acid.

$$2GeH + 2KMnO_4 + 3H_2SO_4 \rightarrow 2GeO_2 + K_2SO_4 + 2MnSO_4 + 4H_2O$$

L. M. Dennis and N. A. Skow, J. Am. Chem. Soc., **52**, 2371 (1930) *1*
16

$GeHCl_3$

H_2O **III-1814**

Germanium chloroform is readily hydrolyzed by water with the formation of hydrated germanous oxide and hydrochloric acid.

$$GeHCl_3 + xH_2O \rightarrow GeO(xH_2O?) + 3HCl$$

L. M. Dennis and H. L. Hunter, J. Am. Chem. Soc., **51**, 1151 (1929) *1*

$GeHCl_3$

H_2O **III-1815**

Germanium chloroform hydrolyzes readily, in oxygen free water, to an orange-colored, insoluble germanous hydroxide. In small amounts of water, a white solid of unspecified composition is formed.

$$GeHCl_3 + 2H_2O \rightarrow Ge(OH)_2 + 3HCl$$

L. M. Dennis, W. K. Orndorff and D. L. Tabern, J. Phys. Chem., **30**, 1054 (1926) *85*

$GeHCl_3$

I **III-1816**

Germanium chloroform is titrated with iodine.

$$GeHCl_3 + 2I \rightarrow GeICl_3 + HI$$

L. M. Dennis, W. R. Orndorff and D. L. Tabern, J. Phys. Chem., **30**, 1049 (1926) *25*

$GeHCl_3$

NaOH **III-1817**

Germanium chloroform in solution is treated with sodium hydroxide.

(a) $GeHCl_3 + 3NaOH \rightarrow Ge(OH)_2 + 3NaCl + H_2O$

(b) $GeHCl_3 + NaOH \rightarrow \underline{GeO} + (NaCl + H_2O + Cl_2)$

A. Hantzsch, Z. anorg. Chem., **30**, 322 (1902)
Ref., Winkler, J. f. prakt. Chem. [2], **34**, 209 (1886) *25*

$GeHCl_3$

Na_2CO_3 **III-1818**

Germanium chloroform is decomposed by a solution of sodium carbonate.

$$2GeHCl_3 + 3Na_2CO_3 + H_2O \rightarrow 2Ge(OH)_2 + 6NaCl + 3CO_2$$

C. Winkler, G. Krüss and L. F. Nilson, Ber., **20**, 1696 (1887) *25*

$GeHCl_3$

O_2 **III-1819**

Above 0°C germanium chloroform is decomposed by dry oxygen; the principal reaction being the formation of the dichloride and tetrachloride.

$$4GeHCl_3 + O_2 \rightarrow 2GeCl_4 + 2GeCl_2 + 2H_2O$$

L. M. Dennis, W. K. Orndorff and D. L. Tabern, J. Phys. Chem., **30**, 1053 (1926) *86*

$GeHCl_3$

Δ **III-1820**

Germanium chloroform decomposes when heated at 140° – 175°.

(a) $$2GeHCl_3 + \Delta \rightarrow 2GeCl_2 + 2HCl$$

(b) $$2GeHCl_3 + \Delta \rightarrow Ge + GeCl_4 + 2HCl$$

D. L. Tabern, L. M. Dennis, W. R. Orndorff, J. Phys. Chem., **30**, 1049 (1926)
Winkler, Küss and Nilson, Ber., **20**, 1696 (1887) *25*

GeH_2

S **III-1821**

Germanous hydride is passed over strongly heated and illuminated sulfur.

$$GeH_2 + 3S \rightarrow GeS_2 + H_2S$$

E. Voegelen, Z. anorg. Chem., **30**, 325 (1902) *25*

GeH_2Cl_2

H_2O **III-1822**

Dichloro monogermane (formed by the action of HCl on monogermane in the presence of a catalyst) reacts with a solution of sodium hydroxide to form the oxide and to evolve hydrogen. Secondary reactions also occur.

$$GeH_2Cl_2 + H_2O \rightarrow GeO + 2HCl + H_2$$

L. M. Dennis and P. R. Judy, J. Am. Chem. Soc., **51**, 2323 (1929) *16*

GeH_2Cl_2

NH_3 **III-1823**

At $-78°$ to $-27°C$, liquid ammonia reacts readily with dichloromonogermane (prepared by method of Dennis and Judy, J. Am. Chem. Soc., **51**, 2321, 1929) to form ammonium chloride and metallic germanium.

$$GeH_2Cl_2 + 2NH_3 \rightarrow Ge + 2NH_4Cl$$

L. M. Dennis and R. W. Work, J. Am. Chem. Soc., **55**, 4488 (1933) *16*

GeH_2Cl_2

NaOH **III-1824**

Germanous oxide is produced when dichloromonogermane is treated with sodium hydroxide.

$$GeH_2Cl_2 + 2NaOH \rightarrow GeO + 2NaCl + H_2O + H_2$$

L. M. Dennis and P. R. Judy, J. Am. Chem. Soc., **51**, 2323 (1929) *1*

GeH_3Cl

H_2O **III-1825**

Monochloromonogermane (formed by the action of HCl on monogermane in the presence of a catalyst) reacts with a solution of sodium hydroxide to form the oxide and to evolve hydrogen. Secondary reactions also occur.

$$GeH_3Cl + H_2O \rightarrow GeO + HCl + 2H_2$$

L. M. Dennis and P. R. Judy, J. Am. Chem. Soc., **51**, 2323 (1929) *16*

GeH_3Cl

NH_3 **III-1826**

When treated with liquid ammonia in excess at −78 to −50°C for two hours, monochloromonogermane (prepared by method of Dennis and Judy, J. Am. Chem. Soc., **51**, 2321, 1929) forms monogermane and monogermanium hydride.

$$3GeH_3Cl + 3NH_3 \rightarrow 3NH_4Cl + GeH_4 + 2GeH$$

L. M. Dennis and R. W. Work, J. Am. Chem. Soc., **55**, 4487 (1933) *16*

GeH_3Cl

NH_3 **III-1827**

Mono-chloromono germane reacts with an excess of ammonia to produce ammonium chloride and a variety of hydrides.

(a) $$GeH_3Cl + NH_3 \rightarrow NH_4Cl + GeH_2$$

(b) $$3GeH_2 \rightarrow GeH_4 + 2GeH$$

L. M. Dennis and R. W. Work, J. Am. Chem. Soc., **55**, 4487 (1933) *1*

GeH_3Cl

NaOH **III-1828**

Germanous oxide is formed when monochloromonogermane reacts with sodium hydroxide.

$$GeH_3Cl + NaOH \rightarrow GeO + 2H_2 + (NaCl)$$

L. M. Dennis and R. W. Judy, J. Am. Chem. Soc., **51**, 2323 (1929) *1*

GeH_3Cl

Decomp. **III-1829**

Monochloromonogermane breaks down slowly at room temperature.

$$2GeH_3Cl \rightarrow GeH_4 + 2HCl + Ge$$

L. M. Dennis and P. R. Judy, J. Am. Chem. Soc., **51**, 2325 (1929) *16*

GeH_3I

HI **III-1830**

When equal volumes of monogermane and hydrogen iodide are mixed and brought into a bulb containing aluminum bromide, the two gases react energetically. Evidence of the formation of GeH_3I and GeH_2I_2 was obtained, but they react with HI to yield GeI_2 as the final product.

(a) $GeH_3I + HI \rightarrow GeH_2I_2 + H_2$

(b) $GeH_2I_2 + HI \rightarrow GeHI_3 + H_2$

(c) $GeHI_3 \rightarrow GeI_2 + HI$

L. M. Dennis and P. R. Judy, J. Am. Chem. Soc., **51**, 2327 (1929) *16*

GeH_4

$AgNO_3$ **III-1831**

Germanic hydride is passed into a solution of silver nitrate.

$$GeH_4 + 4AgNO_3 \rightarrow \underline{Ag_4Ge} + 4HNO_3$$

J. H. Müller and N. H. Smith, J. Am. Chem. Soc., **44**, 1909 (1922) *1*

GeH_4

HBr **III-1832**

Monobromomono germane and dibromomono germane are formed when germane is treated with hydrogen bromide in the presence of aluminum bromide as a catalyst.

$$2GeH_4 + 3HBr \rightarrow GeH_3Br + GeH_2Br_2 + (3H_2)$$

L. M. Dennis and P. R. Judy, J. Am. Chem. Soc., **51**, 2325 (1929) *1*

GeH_4

HCl **III-1833**

Monochloromonogermane and dichloromonogermane are produced when germane reacts with hydrogen chloride in the presence of a catalyst.

$$2GeH_4 + 3HCl \rightarrow GeH_3Cl + GeH_2Cl_2 + (3H_2)$$

L. M. Dennis and P. R. Judy, J. Am. Chem. Soc., **51**, 2322 (1929) *1*

GeH_4

HI

III-1834

Germanous iodide is the final product obtained when hydrogen iodide reacts with germane in the presence of a catalyst.

(a) $2GeH_4 + 3HI \rightarrow GeH_3I + GeH_2I_2 + (3H_2)$

(b) $GeH_3I + HI \rightarrow GeH_2I_2 + H_2$

(c) $GeH_2I_2 + HI \rightarrow GeHI_3 + H_2$

(d) $GeHI_3 \rightarrow GeI_2 + HI$

L. M. Dennis and P. R. Judy, J. Am. Chem. Soc., **51**, 2327 (1929) *1*

GeH_4

Na

III-1835

Sodium trihydrogermanide is produced quantitatively when mono germane is passed through a solution of sodium in liquid ammonia.

$$2GeH_4 + 2Na \rightarrow 2NaGeH_3 + H_2$$

C. A. Kraus and G. A. Carney, J. Am. Chem. Soc., **56**, 766 (1934) *1*

GeH_4

S

III-1836

By passing germanium tetrahydride gas over sulfur which had been exposed previously to sunlight germanium disulfide is obtained.

$$GeH_4 + 4S \rightarrow \underline{GeS_2} + 2H_2S$$

E. Voegelen, Z. anorg. Chem., **30**, 325 (1902) *28*

GeH_4

Δ

III-1837

At temperatures above 280°C, germane decomposes to evolve hydrogen.

$$GeH_4 \rightarrow Ge + 2H_2$$

T. R. Hogness and W. C. Johnson, J. Am. Chem. Soc., **54**, 3583 (1932) *16*

GeH_4

Δ **III-1838**

Germanic hydride is heated to 360° or above.

$$GeH_4 + \Delta \rightarrow Ge + 2H_2$$

J. H. Müller and N. H. Smith, J. Am. Chem. Soc., **44** 1909 (1922)
E. Voegelen, Z. anorg. Chem., **30**, 325 (1902) *25*

GeI_2

C_2H_5I **III-1839**

Ethyl germanium triiodide is formed when germanous iodide is heated to 110° in the absence of air with an excess of ethyl iodide.

$$C_2H_5I + GeI_2 \rightarrow C_2H_5GeI_3$$

E. A. Flood, J. Am. Chem. Soc., **55**, 4935 (1933) *1*

GeI_2

HI **III-1840**

Germanium iodoform is produced by the action of hydrogen iodide on GeI_2.

$$GeI_2 + HI \rightarrow GeHI_3$$

F. M. Brewer and L. M. Dennis, J. Phys. Chem., **31**, 1538 (1927) *85*

GeI_2

I_2 **III-1841**

Germanous iodide reacts with iodine.

$$GeI_2 + I_2 \rightarrow GeI_4$$

F. M. Brewer, J. Phys. Chem., **31**, 1817 (1927) *25*

GeI_2

NH_3 **III-1842**

Germanous iodide (prepared by the reaction between germanous sulfide and concentrated hydrogen iodide solution) reacts with ammonia gas at −33.5°C, in a nitrogen atmosphere to produce germanous imide.

$$GeI_2 + 3NH_3 \rightarrow GeNH + 2NH_4I$$

W. C. Johnson, G. H. Morey and A. E. Kott, J. Am. Chem. Soc., **54**, 4280 (1932) *16*

GeI_2

Decomp. **III-1843**

In the formation of GeI_2 from the reaction of monogermane and hydrogen iodide some GeI_4 is present as a result of the dissociation of the di-iodide.

$$2GeI_2 \rightarrow GeI_4 + Ge$$

L. M. Dennis and P. R. Judy, J. Am. Chem. Soc., **51**, 2327 (1929) *16*

GeI_2

Δ **III-1844**

Germanous iodide is heated to 240–265°.

$$2GeI_2 + \Delta \rightarrow Ge + GeI_4$$

F. M. Brewer, J. Phys. Chem., **31**, 1817 (1927) *25*

GeI_4

$C_2H_5NH_2$ **III-1845**

A substituted germanium imide is produced when germanic iodide is subjected to ethyl amine in carbon tetra chloride solution.

$$GeI_4 + 6C_2H_5NH_2 \rightarrow Ge(NC_2H_5)_2 + 4C_2H_5NH_3I$$

W. C. Johnson and A. E. Sidwell, J. Am. Chem. Soc., **55**, 1887 (1933) *1*

GeI_4

H_2O **III-1846**

Germanic iodide is treated with water.

$$GeI_4 + 2H_2O \rightarrow \underline{GeO_2} + (4HI)$$

L. M. Dennis and F. E. Hance, J. Am. Chem. Soc., **44**, 2854 (1922) *1*

GeI_4

NH_3 **III-1847**

Germanium imide and ammonium iodide are formed when germanic iodide reacts with liquid ammonia.

$$GeI_4 + 6NH_3 \rightarrow Ge(NH)_2 + 4NH_4I$$

W. C. Johnson and A. E. Sidwell, J. Am. Chem. Soc., **55**, 1886 (1933) *1*
16

GeI_4

III-1848

Δ

Germanic iodide is heated to 440°

$$GeI_4 + \Delta \rightarrow GeI_2 + I_2$$

L. M. Dennis and F. E. Hance, J. Am. Chem. Soc., **44**, 2854 (1922) *1*

GeNH

III-1849

H_2O

Germanous imide is decomposed by moisture.

$$GeNH + 2H_2O \rightarrow Ge(OH)_2 + NH_3$$

W. C. Johnson, G. H. Morey, and A. E. Kott, J. Am. Chem. Soc., **54**, 4282 (1932) *1* *16*

GeNH

III-1850

Δ

Germanous imide decomposes into germanous nitride and ammonia when heated for several hours in an electric furnace at 250–300°.

$$3GeNH \rightarrow Ge_3N_2 + NH_3$$

W. C. Johnson and G. H. Ridgely, J. Am. Chem. Soc., **56**, 2396 (1934) *1*

$Ge(NH)_2$

III-1851

Δ

Germanic nitride is formed by the thermal decomposition of germanic imide.

$$3Ge(NH)_2 \rightarrow Ge_3N_4 + (2NH_3)$$

R. Schwarz and P. W. Schenk, Ber., **63**, 296 (1930)
W. C. Johnson and G. H. Ridgely, J. Am. Chem. Soc., **56**, 2395 (1934) *1*

GeO

III-1852

HCl

Germanous oxide is dissolved by hydrochloric acid.

$$GeO + 2HCl \rightarrow GeCl_2 + H_2O$$

Winkler, J. f. prakt. Chem. [2], **34**, 209 (1886) *25*

GeO

KCl

H_2F_2 **III-1853**

Potassium germanium fluoride is prepared by adding a concentrated potassium chloride solution to a solution of germanium oxide in hydrofluoric acid.

$$2GeO + 4KCl + 6H_2F_2 \rightarrow 2K_2GeF_6 + (2H_2O + 4HCl + 2H_2)$$

Winkler, J. f. prakt. Chem., **36**, 188 (1887)
Ref., J. H. K., Am. Chem. J., **10**, 246 (1888) *1*

$Ge(OH)_2$

HI **III-1854**

Germanous hydroxide reacts with hydrogen iodide below 40°.

$$Ge(OH)_2 + 2HI \rightarrow \underline{GeI_2} + 2H_2O$$

F. M. Brewer and L. M. Dennis, J. Phys. Chem., **31**, 1527 (1927) *25*

$Ge(OH)_2$

NaOH **III-1855**

Germanous hydroxide reacts with sodium hydroxide.

$$Ge(OH)_2 + NaOH \rightarrow NaHGeO_3 + (H_2)$$

A. Hantzsch, Z. anorg. Chem., **30**, 322 (1902) *25*

$Ge(OH)_2$

NaOH **III-1856**

Germanous hydroxide dissolves in excess of sodium hydroxide.

$$Ge(OH)_2 + NaOH \rightarrow NaHGeO_2 + H_2O$$

A. Hantzsch, Z. anorg. Chem., **30**, 317 (1920) *25*

$Ge(OH)_2$

Decomp. **III-1857**

Germanous hydroxide decomposes.

$$Ge(OH)_2 \rightarrow GeO + H_2O$$

Winkler, J. f. prakt. Chem. [2], **34**, 209 (1886) *25*

$Ge(OH)_4$

III-1858

H_2S

$KC_2H_3O_2$

Germanium hydroxide, in acetate-buffered media, may be quantitatively converted to potassium thiogermanate by treatment with hydrogen sulfide or potassium sulfide.

$$2Ge(OH)_4 + 5H_2S + 2KC_2H_3O_2 \rightarrow K_2Ge_2S_5 + 2HC_2H_3O_2 + 8H_2O$$

Hobart H. Willard and C. W. Zuehlke, Anal. Ed., **16**, 322 (1944) *44*

GeO_2

III-1859

C

Metallic germanium may be produced in 90% yield by reduction with carbon under a sodium chloride flux.

$$GeO_2 + 2C \rightarrow Ge + 2CO$$

K. M. Tressler and L. M. Dennis, J. Phys. Chem., **31**, 1432 (1927) *85*

GeO_2

III-1860

C

Germanium dioxide is reduced by carbon.

$$GeO_2 + 2C \rightarrow Ge + 2CO$$

Winkler, Ber., **24**, 891 (1891) *25*

GeO_2

III-1861

$COCl_2$

Germanium dioxide is heated to dull redness and a stream of carbonyl chloride passed over it.

$$GeO_2 + 2COCl_2 \rightarrow GeCl_4 + 2CO_2$$

W. Pugh and J. S. Thomas, J. Chem. Soc. (London) p. 1051 (1926) *25*

GeO_2

CaF_2

III-1862

Germanium dioxide is heated with calcium fluoride in the presence of sulfuric acid.

$$GeO_2 + 2CaF_2 + 2H_2SO_4 \rightarrow GeF_4 + 2CaSO_4 + 2H_2O$$

Winkler, J. f. prakt. Chem. [2], **36**, 194 (1887)

25

GeO_2

HBr

III-1863

Germanium tetrabromide is formed when germanium dioxide is suspended in a 48% solution of hydrobromic acid and hydrogen bromide passed into the solution slightly below the boiling point of hydrobromic acid.

$$GeO_2 + 4HBr \rightarrow GeBr_4 + 2H_2O$$

A. W. Laubengayer and P. L. Brandt, J. Am. Chem. Soc., **54**, 622 (1932)

1

GeO_2

HCl

III-1864

Germanium tetrachloride is formed when germanium dioxide is treated with hydrochloric acid.

$$GeO_2 + 4HCl \rightarrow GeCl_4 + 2H_2O$$

D. L. Tabern, W. R. Orndorff and L. M. Dennis, J. Am. Chem. Soc., **47**, 2039 (1925)

1

GeO_2

HF

III-1865

When germanium oxide is dissolved in hydrofluoric acid and the solution evaporated trihydrated germanium fluoride forms as a crystalline deliquescent mass.

(a) $$GeO_2 + 4HF \rightarrow GeF_4 + 2H_2O$$

(b) $$GeF_4 + 3H_2O \rightarrow GeF_4 \cdot 3H_2O$$

P. T. Cleve, Sv. Kem. Tidsk., **1**, 39 (1889)

10

GeO_2

HI **III-1866**

Red crystals of germanium tetra iodide are formed when germanium dioxide reacts with hydriodic acid at 160°.

$$GeO_2 + 4HI \rightarrow GeI_4 + 2H_2O$$

A. W. Laubengayer and P. L. Brandt, J. Am. Chem. Soc., **54**, 622 (1932) *1*

GeO_2

H_2 **III-1867**

Germanium dioxide is reduced to germanium by hydrogen at 550–600°C.

$$GeO_2 + 2H_2 \rightarrow Ge + 2H_2O$$

L. M. Dennis and F. E. Hance, J. Am. Chem. Soc., **44**, 299 (1922) *1*

GeO_2

H_2 **III-1868**

Germanium dioxide is reduced by hydrogen when heated to dull redness.

$$GeO_2 + 2H_2 \rightarrow Ge + 2H_2O$$

C. Winkler, Ber., **24**, 891 (1891)
Ref., Paneth, Schmidt, Hebbel, Ber., **55**, 2615 (1922) *25*

GeO_2

H_2F_2 **III-1869**

Germanium dioxide dissolves in a 20% solution of hydrogen fluoride.

(a) $$GeO_2 + 2H_2F_2 \rightarrow GeF_4 + 2H_2O$$

(b) $$GeO_2 + 3H_2F_2 \rightarrow H_2GeF_6 + 2H_2O$$

G. Krüss and L. F. Nilson, Ber., **20**, 1696 (1887) *25*

GeO_2

H_2F_2 **III-1870**

Germanium tetrafluoride is formed when germanium dioxide is treated with hydrofluoric acid.

$$GeO_2 + 2H_2F_2 \rightarrow GeF_4 + (2H_2O)$$

C. Winkler, J. f. prakt. Chem., **36**, 188 (1887)
Ref., J. H. K., Am. Chem. J., **10**, 246 (1888) *1*

GeO_2

H_2F_2 **III-1871**

Fluogermanic acid is produced when germanium dioxide is treated with hydrofluoric acid.

$$GeO_2 + 3H_2F_2 \rightarrow H_2GeF_6 + (2H_2O)$$

L. M. Dennis and Staneslow, J. Am. Chem. Soc., **55**, 4392 (1933) *1*

GeO_2

H_2F_2 **III-1872**

$BaCl_2$

Germanium dioxide dissolved in hydrofluoric acid is treated with barium chloride.

$$GeO_2 + 3H_2F_2 + BaCl_2 \rightarrow BaGeF_6 + 2HCl + 2H_2O$$

L. M. Dennis, Z. anorg. Chem., **174**, 102 (1928) *25*

GeO_2

H_2F_2 **III-1873**

K_2CO_3

Potassium fluogermanate precipitates when germanium dioxide is dissolved in pure hydrofluoric acid and the resulting hot solution treated with a little more than the calculated amount of potassium carbonate.

$$GeO_2 + 3H_2F_2 + K_2CO_3 \rightarrow \underline{K_2GeF_6} + (3H_2O + CO_2)$$

J. H. Müller, J. Am. Chem. Soc., **43**, 1089 (1921) *1*

GeO_2

III-1874

H_2S

When hydrated germanium oxide is suspended in absolute alcohol and treated with hydrogen sulfide, thiogermanic acid is formed.

$$2GeO_2 + 5H_2S \rightarrow H_2Ge_2S_5 + 4H_2O$$

H. H. Willard and C. W. Zuehlke, J. Am. Chem. Soc., **65**, 1888 (1943) *16*

GeO_2

III-1875

H_2S

Germanium dioxide in H_2SO_4 is treated with hydrogen sulfide.

$$GeO_2 + 2H_2S \rightarrow \underline{GeS_2} + 2H_2O$$

C. Winkler, J. f. prakt. Chem. [2], **36**, 194 (1887) *25*

GeO_2

III-1876

H_2S

CH_3COOK

When hydrogen sulfide is added to a suspension of hydrated germanium dioxide in glacial acetic acid and potassium acetate, a colorless viscous solution of potassium thiogermanate is formed.

$$2GeO_2 + 5H_2S + 2CH_3COOK \rightarrow K_2Ge_2S_5 + 2CH_3COOH + 4H_2O$$

H. H. Willard and C. W. Zuehlke, J. Am. Chem. Soc., **65**, 1888 (1943) *16*

GeO_2

III-1877

H_2Se

Germanium dioxide in 6*N* hydrochloric acid solution is reduced by hypophosphorous acid, 70–75% of acid is neutralized by ammonium hydroxide and germanium monoselenide is precipitated by hydrogen selenide in an atmosphere of carbon dioxide as a brown solid.

(a) $(GeO_2 + 4HCl \rightarrow GeCl_4 + 2H_2O)$

(b) $(GeCl_4 + H_3PO_2 + H_2O \rightarrow GeCl_2 + H_3PO_3 + 2HCl)$

(c) $(GeCl_2 + H_2Se \rightarrow \underline{GeSe} + 2HCl)$

B. N. Ivanoff-Emin, J. Gen. Chem. (USSR), **10**, 1813 (1940) *60*

GeO_2

KCN
C

III-1878

Germanium dioxide is mixed with an excess of potassium cyanide and charcoal and the mixture heated to about 1000°.

(a) $4GeO_2 + 2KCN + 2C \rightarrow 4Ge + (K_2CO_3 + 2NO + 3CO)$

(b) $4GeO_2 + 2KCN + C \rightarrow 3Ge + K_2GeO_3 + (2NO + 3CO)$

J. S. Thomas and W. Pugh, J. Chem. Soc. (London), **125**, 824 (1924) *25*

GeO_2

KOH

III-1879

By treating germanium dioxide with a solution of potassium hydroxide, potassium germanate is formed.

$$GeO_2 + 2KOH \rightarrow K_2GeO_3 + H_2O$$

H. H. Willard and C. W. Zuehlke, J. Am. Chem. Soc., **65**, 1888 (1943) *16*

GeO_2

Mg

III-1880

Germanium dioxide is reduced by magnesium when carefully heated.

(a) $GeO_2 + Mg \rightarrow GeO + MgO$

(b) $GeO_2 + 2Mg \rightarrow Ge + 2MgO$

C. Winkler, Ber., **24**, 891 (1891) *25*

GeO_2

$MgCl_2$
$Mg(OH)_2$

III-1881

Germanium dioxide dissolved in a slightly ammoniacal solution is treated with magnesia mixture.

$$GeO_2 + MgCl_2 + Mg(OH)_2 \rightarrow \underline{Mg_2GeO_4} + 2HCl)$$

J. H. Müller, J. Am. Chem. Soc., **44**, 2494 (1922) *1*

GeO_2

III-1882

NH_3

Ammonia reacts slowly with germanic oxide at 700°C and much more rapidly at 750°C. The germanium is reduced to the metallic form and excess ammonia forms germanic nitride.

$$3GeO_2 + 4NH_3 \rightarrow Ge_3N_4 + 6H_2O$$

W. C. Johnson, J. Am. Chem. Soc., **52**, 5162 (1930) *16*

GeO_2

III-1883

Na-Hg

A 3% sodium amalgam reduces germanium dioxide to germane in alkaline solution.

$$GeO_2 + 8H \rightarrow GeH_4 + 2H_2O$$

S. A. Coase, Analyst, **59**, 747 (1934) *40*

GeO_2

III-1884

ε

Germanium dioxide is reduced to germane when a current is passed through a solution alkaline with sodium hydroxide using nickel electrodes.

$$GeO_2 + 8H \rightarrow GeH_4 + 2H_2O$$

S. A. Coase, Analyst, **59**, 462 (1934) *40*

GeO_2

III-1885

ε

Germanium dioxide is dissolved in fused cryolite and electrolyzed using a graphite cathode.

$$GeO_2 \rightarrow \underline{Ge} + O_2$$

L. M. Dennis Z., anorg. Chem., **174**, 102 (1928) *25*

GeS

HI **III-1886**

Germanous iodide is prepared by the action of concentrated hydriodic acid on germanous sulfide.

$$GeS + 2HI \rightarrow GeI_2 + (H_2S)$$

W. C. Johnson, G. H. Morey and A. E. Kott, J. Am. Chem. Soc., **54**, 4279 (1932) *1*

GeS

O_2 **III-1887**

Germanous sulfide is heated in air to bright redness.

$$GeS + 2O_2 \rightarrow GeO_2 + SO_2$$

C. Winkler, Ber., **24**, 891 (1891) *26*

GeS_2

HNO_3 **III-1888**

Germanic sulfide is treated with nitric acid.

$$GeS_2 + 4HNO_3 \rightarrow GeO_2 + (2SO_2 + 2H_2O + 4NO)$$

L. M. Dennis and W. C. Johnson, J. Am. Chem. Soc., **45**, 1380 (1923) *1*

GeS_2

H_2 **III-1889**

The optimum temperature for the reduction of germanium disulfide to the mono-sulfide is 480°C.

(a) $$GeO_2 + 4HCl \rightarrow GeCl_4 + 2H_2O$$

(b) $$GeCl_4 + 2H_2S \rightarrow GeS_2 + 4HCl$$

(c) $$GeS_2 + H_2 \rightarrow GeS + H_2S$$

L. M. Dennis and S. M. Joseph, J. Phys. Chem., **31**, 1716 (1927) *85*

GeS_2

III-1890

H_2O

Freshly precipitated germanium sulfide reacts with water forming germanium oxide and hydrogen sulfide, particularly if heated.

$$GeS_2 + 2H_2O \rightarrow GeO_2 + 2H_2S$$

P. T. Cleve, Sv. Kem. Tidsk., **1**, 39 (1889) *10*

GeS_2

III-1891

H_2O

Germanic sulfide can be conveniently transformed to the dioxide by simple hydrolysis. This decomposition is effected by injecting a current of steam into a water suspension of the sulfide.

$$(GeS_2 + 2H_2O \rightarrow GeO_2 + 2H_2S)$$

Müller and Eisner, Anal. Ed., **4**, 134 (1932) *114*

GeS_2

III-1892

H_2O_2

An ammoniacal solution of germanic sulfide is oxidized by hydrogen peroxide.

$$GeS_2 + 6H_2O_2 \rightarrow GeO_2 + (2SO_2 + 6H_2O)$$

W. C. Johnson and L. M. Dennis, J. Am. Chem. Soc., **47**, 790 (1925) *1*

GeS_2

III-1893

$HgCl_2$

Germanium tetrachloride is formed when a mixture of germanium sulfide and mercuric chloride is gently heated.

$$GeS_2 + 2HgCl_2 \rightarrow GeCl_4 + (2HgS)$$

Winkler, J. f. prakt. Chem., **36**, 188 (1887)
Ref., J. H. K., Am. Chem. J., **10**, 246 (1888) *1*

Ge_3N_2

H_2 **III-1894**

Passing hydrogen gas over germanous nitride at 500–600° reduces the nitride to metallic germanium.

$$Ge_3N_2 + 3H_2 \rightarrow 3Ge + 2NH_3$$

W. C. Johnson and G. H. Ridgely, J. Am. Chem. Soc., **56**, 2396 (1934) *1*

Ge_3N_2

Δ **III-1895**

Germanous nitride decomposes into its elements at 850°.

$$Ge_3N_2 \rightarrow 3Ge + N_2$$

W. C. Johnson and G. H. Ridgely, J. Am. Chem. Soc., **56**, 2396 (1934) *1*

Ge_3N_4

Cl_2 **III-1896**

At 600–700°C the reaction between germanic nitride and chlorine gas proceeds rapidly.

$$Ge_3N_4 + 6Cl_2 \rightarrow 3GeCl_4 + 2N_2$$

W. C. Johnson, J. Am. Chem. Soc., **52**, 5163 (1930) *16*

Ge_3N_4

H_2 **III-1897**

When germanic nitride is heated to 700°C in a stream of hydrogen gas, reaction is extremely rapid.

$$Ge_3N_4 + 6H_2 \rightarrow 3Ge + 4NH_3$$

W. C. Johnson, J. Am. Chem. Soc., **52**, 5162 (1930) *16*

Ge_3N_4

Δ **III-1898**

At high temperatures, germanic nitride evolves nitrogen.

$$Ge_3N_4 \rightarrow 3Ge + 2N_2$$

G. H. Morey and W. C. Johnson, J. Am. Chem. Soc., **54**, 3603 (1932) *16*

Ge_3N_4 **III-1899**

Δ

Germanic nitride decomposes into its elements above 900°.

$$Ge_3N_4 \rightarrow 3Ge + 2N_2$$

W. C. Johnson and G. H. Ridgely, J. Am. Chem. Soc., **56**, 2395 (1934) *1*

H_2GeF_6 **III-1900**

Cs_2F_2

Fluogermanic acid, obtained by $GeO_2 + H_2F_2$ is treated with cesium fluoride.

(a) $$2GeO_2 + 5H_2F_2 \rightarrow H_2GeF_6 + GeF_4 + 4H_2O$$

(b) $$H_2GeF_6 + Cs_2F_2 \rightarrow \underline{Cs_2GeF_6} + H_2F_2$$

Skrabal and Gruber, Monatsh, **38**, 19 (1917) *30*

H_2GeF_6 **III-1901**

K_2F_2

Fluogermanic acid in acid solution reacts with potassium fluoride.

$$H_2GeF_6 + K_2F_2 \rightarrow \underline{K_2GeF_6} + H_2F_2$$

G. Krüss and L. F. Nilson, Ber., **20**, 1696 (1887) *25*

H_2GeF_6 **III-1902**

NH_2OH

Hydroxylamine fluogermanate is formed when a cooled solution of fluogermanic acid is treated with either hydroxylamine or the hydrochloride.

$$H_2GeF_6 + 2NH_2OH + 2H_2O \rightarrow (NH_2OH)_2 \cdot H_2GeF_6 \cdot 2H_2O$$

L. M. Dennis and Staneslow, J. Am. Chem. Soc., **55**, 4392 (1933) *1*

H_2GeF_6

NH_3 **III-1903**

Ammonium fluogermanate is precipitated when pure ammonia gas is passed through a cooled solution of fluogermanic acid.

$$H_2GeF_6 + 2NH_3 \rightarrow \underline{(NH_4)_2GeF_6}$$

L. M. Dennis and Staneslow, J. Am. Chem. Soc., **55**, 4392 (1933) *1*

K_2GeF_6

H_2SO_4 **III-1904**

A mixture of potassium fluogermanate and sulfuric acid is heated till the excess of sulfuric acid is removed.

$$K_2GeF_6 + H_2SO_4 \rightarrow GeF_4 + (K_2SO_4 + H_2F_2)$$

C. Winkler, J. f. prakt. Chem. [2], **36**, 194 (1887) *25*

K_2GeO_3

CH_3COOH **III-1905**

From a solution of potassium germanate hydrated germanium dioxide may be precipitated by addition of glacial acetic acid.

$$K_2GeO_3 + 2CH_3COOH \rightarrow GeO_2 + 2CH_3COOK + H_2O$$

H. H. Willard and C. W. Zuehlke, J. Am. Chem. Soc., **65**, 1888 (1943) *16*

$K_2Ge_2S_5$

I_2 **III-1906**

Excess hydrogen sulfide may be removed from a potassium thiogermanate solution by passing a rapid stream of carbon dioxide through the solution, after which the sulfide ion is determined by treatment with an excess of standard iodine solution and back titrated with sodium thiosulfate solution.

$$K_2Ge_2S_5 + 5I_2 + 8H_2O \rightarrow 5S + 2Ge(OH)_4 + 8HI + 2KI$$

Hobart H. Willard and C. W. Zuehlke, Anal. Ed., **16**, 322 (1944) *44*

Mg_2Ge

III-1907

HCl

Magnesium germanide is treated with hydrochloric acid at a low temperature.

(a) $Mg_2Ge + 4HCl \rightarrow 2MgCl_2 + GeH_4$

(b) $2Mg_2Ge + 8HCl \rightarrow 4MgCl_2 + Ge_2H_6 + H_2$

(c) $3Mg_2Ge + 12HCl \rightarrow 6MgCl_2 + Ge_3H_8 + 2H_2$

L. M. Dennis, R. B. Corey and R. W. Moore, J. Am. Chem. Soc., **46**, 657 (1924) *1*

$NaGeH_3$

III-1908

O_2

When oxygen is introduced into a solution of sodium trihydrogermanide in liquid ammonia, a white, solid precipitate is formed.

$$NaGeH_3 + 2O_2 \rightarrow \underline{NaO \cdot Ge(OH)_3}$$

C. A. Kraus and E. S. Carney, J. Am. Chem. Soc., **56**, 767 (1934) *16*

$NaGeH_3$

III-1909

Δ

With rising temperature sodium trihydrogermanide decomposes to evolve hydrogen. The rate of evolution of hydrogen is greatest at around 125°C, although hydrogen is slowly evolved at temperatures up to 360°C.

$$2NaGeH_3 \rightarrow 2NaGe + 3H_2$$

C. A. Kraus and E. S. Carney, J. Am. Chem. Soc., **56**, 767 (1934) *16*

$NaHGeO_3$

III-1910

H_2O

Sodium bigermanate hydrolyzes to yield sodium hydroxide and germanic acid.

$$NaHGeO_3 + H_2O \rightarrow NaOH + H_2GeO_3$$

C. E. Gulezian and J. H. Müller, J. Am. Chem. Soc., **54**, 3153 (1932) *16*

Au

$AuBr_2$ **III-1911**

The electrochemical behavior of gold in hydrogen bromide solutions is studied and discussed.

$$3AuBr_2 \rightarrow 2Au + AuBr_4^{++} + 2Br^-$$

G. Grube and T. Morita, Z. Elektrochem., **38**, 117 (1932) *86*

Au

Br_2 **III-1912**

Bromine reacted with gold, forming auric bromide.

$$2Au + 3Br_2 \rightarrow 2AuBr_3$$

J. W. Mallet, Trans. Roy. Soc. (London), **180**, p. 412 (1889) *105*

Au

Cd **III-1913**

A careful analysis of the freezing point data of Heycock and Neville, J. Chem. Soc., **59**, 936 (1891), for the system gold-cadmium-tin shows that the two following compounds are formed as stable phases.

(a) $$Au + Cd \rightarrow \underline{AuCd}$$

(b) $$Au + 3Cd \rightarrow \underline{AuCd_3}$$

E. S. Shepherd, J. Phys. Chem., **8**, 92 (1904) *7*

Au

Cl **III-1914**

Gold can be dissolved if it is made anode in hydrochloric acid electrolysis:

$$Au + 3Cl(atoms) + HCl \rightarrow HAuCl_4$$

Emil Wohlwill, Z. Elektrochem., **4**, 383 (1898) *86*

Au

III-1915

Cl_2

Auric chloride can be prepared by the direct action of chlorine on metallic gold, provided that the temperature is kept high enough.

$$(2Au + 3Cl_2 \rightarrow 2AuCl_3)$$

Geohand Krüss, Ann., **238**, 30 (1887) *17*
Ref., J. W. Mallet, Am. Chem. J., **12**, 77 (1890) *105*

Au

III-1916

Cl_2

Gold is attacked by free chlorine at 200°C. At 300–350° the chloride decomposes back into gold and chlorine.

$$(3Cl_2 + 2Au \rightleftharpoons 2AuCl_3)$$

A. Jaquerod and M. Tourpaian, J. Chim. phys., **11**, 3 (1913) *69*

Au

III-1917

Cl_2

HCl

Chlorauric acid may be prepared by treating pure gold in hydrochloric acid suspension by chlorine. Evaporation of the solution obtained yields the trihydrate.

$$2Au + 2HCl + 3Cl_2 + 6H_2O \rightarrow 2HAuCl_4 \cdot 3H_2O$$

J. Jacobsen, Compt. rend., **146**, 1214 (1908) *88*

Au

III-1918

HCl

HNO_3

Gold was dissolved in aqua regia, forming auric chloride.

$$2Au + 9HCl + 3HNO_3 \rightarrow 2AuCl_3 + (3NOCl + 6H_2O)$$

W. C. Roberts-Austin, Trans. Roy. Soc. (London), **179**, 349 (1888) *105*

Au-Pt alloy

HCl **III-1919**

HNO_3

Aqua regia was added to gold 10 parts platinum 90 parts alloy, forming auric chloride; it was then reduced to metallic gold by oxalic acid for the purpose of analysis.

(a) $2Au + 9HCl + 3HNO_3 \rightarrow 2AuCl_3 + 3NOCl + 6H_2O$

(b) $2AuCl_3 + 3H_2C_2O_4 \rightarrow 2Au + (6CO_2 + 6HCl)$

Edward Matthey, Trans. Roy. Soc. (London), **183**, 637 (1892) *105*

Au

HCl **III-1920**

HNO_3

When gold dissolves in aqua regia, it goes entirely into the auric state.

$$Au + 4HCl + HNO_3 \rightarrow HAuCl_4 + 2H_2O + NO$$

Victor E. Herschlag, Anal. Ed., **13**, 561 (1941) *44*

Au

HCl **III-1921**

HNO_3

An intermediate gold oxide is obtained when gold is dissolved incompletely in aqua regia in which hydrochloric acid is in excess.

$$2Au + 3HCl + 3HNO_3 \rightarrow Au_2O_2 + (3NOCl + 3H_2O + O)$$

Prat, Compt. rend., **70**, 843
Ref., J. Am. Chem. Soc., **25**, 1136 (1903) *1*

Au

III-1922

HCl

HNO_3

SO_2

Impure gold is dissolved in aqua regia, diluted with water to precipitate silver chloride that may be present and the solution filtered. The filtrate is concentrated on the water bath with additions of hydrochloric acid until all of the nitric acid is removed. Hydrochlorauric acid may precipitate during this procedure, but may be redissolved by the addition of water. The solution so obtained is diluted with water and sulfur dioxide is passed into it until precipitation is complete. The precipitated gold is filtered off and washed with water. It is then dried at 170-180°C.

(a) $Au + HNO_3 + 4HCl \rightarrow HAuCl_4 + NO + 2H_2O$

(b) $2HAuCl_4 + 6H_2O + 3SO_2 \rightarrow 2Au + 3H_2SO_4 + 8HCl$

G. Kruss, Ann., **238**, 43 (1887) *20*

Au

III-1923

HI

When hydrogen iodide is directed into ether containing gold leaf, some gold is dissolved.

$$2Au + 6HI \rightarrow 2AuI_3 + 3H_2$$

J. Nickles, Compt. rend., **62**, 756 (1866) *29*

Au

III-1924

HI

Fe_2O_3

In the presence of the higher oxides of some metals such as iron, manganese, and bismuth, hydriodic acid reacts with gold to form aurous iodide.

$$2Au + Fe_2O_3 + 4HI \rightarrow 2AuI + (FeI_2 + FeO + 2H_2O)$$

J. Nickles, Compt. rend., **62**, 756 (1866) *29*

Au

HNO_3 **III-1925**

HCl

Cesium auric chloride is formed when gold is dissolved in aqua regia and a hot solution of cesium nitrate added.

(a) $2Au + 3HNO_3 + 3HCl \rightarrow 2HAuCl_4 + 3HNO_2 + HCl + 3H_2O$

(b) $HAuCl_4 + CsNO_3 \rightarrow \underline{CsAuCl_4} + (HNO_3)$

L. McCluskey and L. Echelberger, J. Am. Chem. Soc., **48**, 137 (1926) *1*

Au

H_2O_2 **III-1926**

Gold reacts with and dissolves in an acid solution of hydrogen peroxide.

(a) $2Au + H_2O_2 \rightarrow Au_2O + (H_2O)$

(b) $Au_2O + H_2O_2 \rightarrow 2Au + H_2O + O_2$

T. Fairley,
Ref., R. Gerstl., Ber., **9:1**, 948 (1876) *26*

Au

H_2SeO_4 **III-1927**

Gold leaf dissolves in concentrated selenic acid at 300° with the formation of auric selenate.

$$2Au + 4H_2SeO_4 \rightarrow Au_2(SeO_4)_3 + SeO_2 + (2H_2O + 2H_2)$$

V. Lenher, J. Am. Chem. Soc., **24**, 355 (1902) *1*

Au

III-1928

I_2

Cl_2

A good yield of lemon-yellow, crystalline, aurous iodide is obtained by shaking precipitated gold with a solution of iodine in potassium iodide for sixteen to twenty-four hours at 25°C (a), aurous chloride may be prepared by dechlorination of auric chloride (b), at room temperatures aurous chloride slowly decomposes (c), aurous bromide behaves similarly.

(a) $$2Au + I_2 \rightarrow 2AuI$$

(b) $$AuCl_3 \rightarrow AuCl + Cl_2 \text{ (120–200°C)}$$

(c) $$3AuCl \rightarrow AuCl_3 + 2Au$$

F. H. Campbell, Trans. Faraday Soc., **3**, 104 (1907) *85*

Au

III-1929

KBr

Br_2

Twenty parts of finely divided gold and 12.5 parts of dry potassium bromide, (dissolved in 24 times its weight of water) are treated with 30 parts of bromine in a stoppered flask set in a warm place until all of the gold has dissolved. Evaporate the contents of the flask on the water bath to dryness, dissolve residue in water, filter and concentrate until a portion solidifies on a stirring rod. Cool, crystals separate which are recrystallized to remove the potassium bromide. Auric bromide with five H_2O remains.

(a) $$2Au + 3Br_2 + 2KBr \rightarrow 2KAuBr_4$$

(b) $$KAuBr_4 + 5H_2O \rightarrow KBr + AuBr_3 \cdot 5H_2O$$

P. Schottländer, Ann., **217**, 314 (1883) *20*

Au

KCN **III-1930**

A .25% solution of potassium (or sodium or calcium) cyanide dissolves gold from ore in presence of excess air.

$$4Au + 8KCN + O_2 + 2H_2O \rightarrow 4KAu(CN)_2 + 4KOH$$

C. Spearman, Can. Chem. & Met., **9**, 56 (1925) *15*

Au

KCN **III-1931**

Gold is dissolved in alkali cyanide according to the equation:

$$8KCN + Au + O_2 + 2H_2O \rightarrow 4KAu(CN)_2 + 4KOH$$

Elsner, J. f. prakt. Chem., **37**, 441 (1888)
Ref., J. J. Andrejew, Z. Elektrochem., **19**, 667 (1913) *86*

Au

KCN **III-1932**

O_2

Finely divided gold suspended in potassium cyanide and boiled in presence of air forms the double potassium aurous cyanide and potassium hydroxide.

$$4KCN + 2Au + 2H_2O \rightarrow 2KCN \cdot AuCN + 2KOH + (H_2)$$

C. G. Lindbom, Acta Univ. Lund., **12**, 2, No. 6, 10 (1876) *10*

Au

KCN **III-1933**

O_2

Gold can be dissolved in potassium cyanide if the cyanide solution is exposed to air to dissolve oxygen.

$$4Au + 8KCN + O_2 + 2H_2O \rightarrow 4(AuCN \cdot KCN) + 4KOH$$

N. S. Keith, Trans. Am. El. Chem. Soc., **3**, 229 (1903) *8*

Au

KCN **III-1934**

O_2

It was found that the amount of gold which could be dissolved in potassium cyanide solution is dependent upon the amount of oxygen present. In a solution which was free from oxyen, the amount of gold which reacted with the potassium cyanide solution was negligible. The fact applies equally well to silver.

(a) $4Au + 8KCN + 2H_2O + O_2 \rightarrow 4AuCN \cdot KCN + 4KOH$

(b) $(4Ag + 8KCN + 2H_2O + O_2 \rightarrow 4AgCN \cdot KCN + 4KOH)$

J. S. Maclaurin, J. Chem. Soc. (London), **67**, 199 (1895) *103*

Au

III-1935

K_2O_4

The compound K_2O_4 formed by burning potassium is a powerful oxidizing agent, converting metallic gold into potassium aurate.

(a) $$2K + 2O_2 \rightarrow K_2O_4$$

(b) $$K_2O_4 + 2Au \rightarrow 2KAuO_2$$

K. A. Hofmann and H. Hiendlmaier, Ber., **39**, 3184 (1906)
Ref., J. Am. Chem. Soc., **29**, 3821 (1907) *1*

Au

III-1936

$MnCl_4$

Gold is dissolved by aqueous or ethereal solutions of manganese, cobalt or nickel perchlorides.

(a) $$2Au + 3MnCl_4 \rightarrow 2AuCl_3 + 3MnCl_2$$

(b) $$Au + 3CoCl_3 \rightarrow AuCl_3 + 3CoCl_2$$

(c) $$Au + 3NiCl_3 \rightarrow AuCl_3 + 3NiCl_2$$

J. Nickles, Compt. rend., **62**, 755 (1866) *29*

Au

III-1937

Na_2O_2

H_2SO_4

KOH

When precipitated gold is added to fused sodium peroxide, sodium aurate is formed. Auric hydroxide is precipitated from the aurate by dilute sulfuric acid.

Potassium aurate is prepared by treating auric hydroxide with potassium hydroxide.

(a) $$2Au + 3Na_2O_2 \rightarrow 2NaAuO_2 + (2Na_2O)$$

(b) $$2NaAuO_2 + H_2SO_4 + 2H_2O \rightarrow \underline{Au_2O_2(OH)_2 \cdot 2H_2O} + (Na_2SO_4)$$

(c) $$Au_2O_2(OH)_2 \cdot 2H_2O + 2KOH \rightarrow 2KAuO_2 + (4H_2O)$$

F. Meyer Compt. rend., **145**, 805 (1907)
Ref., E. Horton, J. Chem. Soc. (London), **94:2**, 47 (1908) *57*

Au

S **III-1938**

If gold is heated in its form of an alloy with some other metal, with sulfur the compound Au_2S is formed along with the sulfide of the alloyed metal.

$$(2(Au,Cu) + 2S \rightarrow Cu_2S + Au_2S)$$

J. S. Maclaurin, J. Chem. Soc. (London), **69:2**, 2, 1270 (1896) *103*

Au

$SOCl_2$ **III-1939**

Gold foil reacts very slowly and incompletely with thionyl chloride, after several days heating at 200° in a sealed tube.

$$2Au + 4SOCl_2 \rightarrow 2AuCl_3 + (2SO_2 + S_2Cl_2)$$

H. B. North and A. M. Hageman, J. Am. Chem. Soc., **34**, 892 (1912) *1*

Au

SO_2Cl_2 **III-1940**

Powdered gold ore is treated with excess sulfuryl chloride, placed in a sealed tube and the mixture maintained at 160° in an oil bath for many hours. When the reaction is complete red crystals, $AuCl_3$, appear and, on breaking the tube, SO_2 gas escapes.

$$2Au + 3SO_2Cl_2 \rightarrow 2AuCl_3 + 3SO_2$$

H. B. North, Bull. soc. chim. [4], **9**, 647 (1911) *31*

Au

Te **III-1941**

A study of the fusion curve formed by mixtures of gold and tellurium, particularly those of more than 60% gold, shows that the solidification temperature reaches a maximum at 472°, where the compound Au_2Te_4, known as Calaverite, apparently exists. No indication of a compound corresponding to Au_2Te is given.

$$2Au + 4Te \rightarrow Au_2Te_4$$

H. Pélabon, Compt. rend., **148**, 1176–7 (1909) *38*

Au^{+++}

III-1942

H_2

Hydrogen gas precipitates metallic gold from auric solutions.

$$(2Au^{+++} + 3H_2 \rightarrow \underline{2Au} + 6H^+)$$

W. J. Russell, J. Chem. Soc. (London), **27**, 11 (1874) *48*

$AuBr_3$

III-1943

CsBr

A solution of gold bromide will react with a solution of cesium bromide yielding a black precipitate.

$$AuBr_3 + CsBr \rightarrow \underline{CsAuBr_4}$$

H. L. Wells and H. L. Wheeler, Z. anorg. Chem., **2**, 304 (1892) *28*

$AuBr_3$

III-1944

KBr

Auric bromide and potassium bromide reacted, forming potassium auribromide.

$$AuBr_3 + KBr \rightarrow KAuBr_4$$

J. W. Mallet, Trans. Roy. Soc. (London), **180**, 413 (1889) *105*

$AuBr_3$

III-1945

Zn

Auric bromide was treated with zinc, forming gold and zinc bromide.

$$3Zn + 2AuBr_3 \rightarrow 2Au + (3ZnBr_2)$$

J. W. Mallet, Trans. Roy. Soc. (London), **180**, 431 (1889) *105*

AuCl

$Mg(OH)_2$ **III-1946**

HNO_3

80 grams of gold monochloride are decomposed by treating with water and the solution filtered to remove the precipitated gold. The filtrate is heated to boiling and magnesia added while stirring vigorously until the liquid is colorless. Filter and suspend the residue in 1600 cc of water. Add 800 cc of nitric acid (sp. gr. 1.40) and allow to stand 24 hours. Filter and suspend the residue in an equal volume of water and heat to boiling under a reflux condenser for six hours. Filter and wash the residue with hot water. Auryl hydroxide is formed.

(a) $$3AuCl \rightarrow AuCl_3 + 2Au$$

(b) $$2AuCl_3 + 4Mg(OH)_2 \rightarrow Mg(AuO_2)_2 + 3MgCl_2 + 4H_2O$$

(c) $$Mg(AuO_2)_2 + 2HNO_3 \rightarrow 2AuOOH + Mg(NO_3)_2$$

G. Krüss, Ann., 2. **237**, 290 (1887) *20*

AuCOCl

$AuCl_3$ **III-1947**

A benzene solution of aurous chloride carbonyl reduces auric chloride to the aurous form.

$$AuCOCl + AuCl_3 \rightarrow 2AuCl + COCl_2$$

M. S. Kharasch and H. S. Isbell, J. Am. Chem. Soc., **52**, 2922 (1930) *16*

AuCOCl

Br_2 **III-1948**

When a benzene solution of aurous chloride carbonyl is treated with bromine, carbon monoxide is evolved.

$$AuCOCl + Br_2 \rightarrow AuClBr_2 + CO$$

M. S. Kharasch and H. S. Isbell, J. Am. Chem. Soc., **52**, 2922 (1930) *16*

AuCOCl

III-1949

H_2O

A benzene solution of aurous chloride carbonyl will evolve carbon monoxide and dioxide on addition of water.

$$2AuCOCl + H_2O \rightarrow 2Au + CO + CO_2 + 2HCl$$

M. S. Kharasch and H. S. Isbell, J. Am. Chem. Soc., **52**, 2922 (1930) *16*

AuCOCl

III-1950

KI

A benzene solution of aurous chloride carbonyl liberates carbon monoxide when treated with potassium iodide.

$$AuCOCl + KI \rightarrow AuI + CO + KCl$$

M. S. Kharasch and H. S. Isbell, J. Am. Chem. Soc., **52**, 2922 (1930) *16*

AuCOCl

III-1951

PCl_3

When a benzene solution of aurous chloride carbonyl is treated with phosphorus trichloride, carbon monoxide is liberated.

$$AuCOCl + PCl_3 \rightarrow AuCl \cdot PCl_3 + CO$$

M. S. Kharasch and H. S. Isbell, J. Am. Chem. Soc., **52**, 2922 (1930) *16*

AuCOCl

III-1952

Decomp.

Aurous chloride carbonyl (formed by the action of dry carbon monoxide on dry auric chloride) at 120° decomposes into metallic gold, carbon monoxide and phosgene.

$$2AuCOCl \rightarrow 2Au + CO + COCl_2$$

M. S. Kharasch and H. S. Isbell, J. Am. Chem. Soc., **52**, 2920 (1930) *16*

$AuCOCl$

Decomp. **III-1953**

In vacuo, solid aurous chloride carbonyl loses carbon monoxide leaving a residue of aurous chloride.

$$AuCOCl \rightleftharpoons AuCl + CO$$

M. S. Kharasch and H. S. Isbell, J. Am. Chem. Soc., **52**, 2922 (1930) *16*

$AuCl_3$

AgF **III-1954**

Auric hydroxide is precipitated quantitatively along with silver chloride when solutions of silver fluoride and auric chloride are brought together.

$$AuCl_3 + 3AgF + 3H_2O \rightarrow \underline{3AgCl} + \underline{Au(OH)_3} + 3HF$$

S. Benedict and J. F. Snell, J. Am. Chem. Soc., **25**, 1138 (1903) *1*

$AuCl_3$

Ag_2CrO_4 **III-1955**

A solution of auric chromate is obtained by treating an excess of freshly precipitated silver chromate with a solution of auric chloride. On evaporation the solution deposits first gold, and then an acid chromate of gold.

(a) $$2AuCl_3 + 3Ag_2CrO_4 \rightarrow Au_2(CrO_4)_3 + (6AgCl)$$

(b) $$2Au_2(CrO_4)_3 \rightarrow 4Au + (6CrO_3 + 3O_2)$$

(c) $$Au_2(CrO_4)_3 + CrO_3 \rightarrow \underline{Au_2(CrO_4)_3 \cdot CrO_3}$$

N. A. Orloff, Chem. Zeit., **31**, 1182 (1907)
Ref., W. H. Glover, J. Chem. Soc. (London), **94:2**, 48 (1908) *57*

$AuCl_3$

Au_2S_2 **III-1956**

Gold chloride acts on Au_2S_2 in the presence of water to form metallic gold, hydrochloric acid and sulfuric acid.

$$4AuCl_3 + Au_2S_2 + 8H_2O \rightarrow 6Au + 12HCl + 2H_2SO_4$$

U. Antony and A. Lucchesi, Gazz. chim. ital., **19**, 555 (1889) *21*

$AuCl_3$

III-1957

CH_3NH_2

HI

Methyl ammonium auric iodide is precipitated when methyl amine hydroiodide, made by action of methyl amine on hydriodic acid, is treated with auric chloride solution.

(a) $$2CH_3NH_2 + 2HI \rightarrow 2CH_3NH_2HI$$

(b) $$4CH_3NH_2HI + AuCl_3 \rightarrow \underline{CH_3NH_2HI \cdot AuI_3} + (3CH_3NH_2HCl)$$

S. D. Gupta, J. Am. Chem. Soc., **36**, 748 (1914) *1*

$AuCl_3$

III-1958

$(CH_3)_2S$

A flocculent white precipitate is produced when methyl sulfide is treated with a solution of auric chloride.

$$(CH_3)_2S + AuCl_3 \rightarrow \underline{AuCl(CH_3)_2S} + (Cl_2)$$

F. C. Phillips, J. Am. Chem. Soc., **23**, 257 (1901) *1*

$AuCl_3$

III-1959

CO

When dry carbon monoxide is passed over anhydrous auric chloride at a temperature of 100°–130°, a low yield of aurous chloride carbonyl is obtained.

(a) $$AuCl_3 + CO \rightarrow AuCl + COCl_2$$

(b) $$AuCl + CO \rightarrow AuCOCl$$

M. S. Kharasch and H. S. Isbell, J. Am. Chem. Soc., **52**, 2925 (1930) *16*

$AuCl_3$

$C_7H_5NS_2$

III-1960

Gold chloride will be reduced to metallic gold by mercapto benzothiazol.

$$AuCl_3 + C_7H_5NS_2 \rightarrow Au + ?$$

G. Spacu and M. Kuras, Z. anal. Chem., **104**, 88 (1936) *28*

$AuCl_3$

$C_{10}H_7NH_2 \cdot HCl$ **III-1961**

Gold chloride will react with a 0.1% solution of Alpha-naphtylamine hydrochloride yielding a red precipitate.

$$AuCl_3 + C_{10}H_7NH_2 \cdot HCl \rightarrow \underline{AuCl \cdot C_{10}H_7 \cdot NH_2 \cdot HCl} + (Cl_2)$$

H. Holzer and W. Reif, Z. anal. Chem., **92**, 12 (1933) *28*

$AuCl_3$

$CaCO_3$ **III-1962**

Calcite reduces gold chloride at 310° under pressure to metallic gold.

$$2AuCl_3 + 3CaCO_3 \rightarrow 2Au + (3CaCl_2 + 3CO_2 + 3O)$$

H. H. Morris, J. Am. Chem. Soc., **40**, 927 (1918) *1*

$AuCl_3$

CaO_2 **III-1963**

Calcium peroxide precipitates metallic gold from gold solutions.

$$2AuCl_3 + 3CaO_2 \rightarrow \underline{2Au} + (3CaCl_2 + 3O_2)$$

V. Lenher, J. Am. Chem. Soc., **36**, 1424 (1914) *1*

$AuCl_3$

CsCl **III-1964**

Solutions of gold chloride and cesium chloride will react with each other yielding a yellow precipitate.

$$AuCl_3 + CsCl \rightarrow \underline{CsAuCl_4}$$

H. L. Wells and H. L. Wheeler, Z. anorg. Chem., **2**, 304 (1892) *28*

$AuCl_3$

CsI **III-1965**

Cesium auric iodide is precipitated as a black compound when a solution of auric chloride is added to a solution of cesium iodide.

$$AuCl_3 + 4CsI \rightarrow \underline{CsI \cdot AuI_3} + (3CsCl)$$

S. D. Gupta, J. Am. Chem. Soc., **36**, 748 (1914) *1*

$AuCl_3$

III-1966

$FeSO_4$

A solution of ferrous sulfate precipitates gold from a solution of gold chloride.

$$2AuCl_3 + 6FeSO_4 \rightarrow 2Au + 2FeCl_3 + 2Fe_2(SO_4)_3$$

E. G. R. Ardagh, F. S. Seaborne and N. S. Grant, Can. Chem. & Met., **8**, 140 (1924) *15*

$AuCl_3$

III-1967

$FeSO_4$

Metallic gold may be precipitated from an auric chloride solution free from nitric acid with an excess of ferrous sulfate solution or other reducing agent.

$$AuCl_3 + 3FeSO_4 \rightarrow Au + Fe_2(SO_4)_3 + FeCl_3$$

Victor E. Herschlag, Anal. Ed., **13**, 561 (1941) *44*

$AuCl_3$

III-1968

HBr

By repeatedly treating auric chloride with hydrobromic acid, hydrobromoauric acid was formed.

(a) $$AuCl_3 + 3HBr \rightarrow AuBr_3 + (3HCl)$$

(b) $$AuBr_3 + HBr \rightarrow HAuBr_4$$

J. W. Mallet, Trans. Roy. Soc. (London), **180**, 406 (1889) *105*

$AuCl_3$

III-1969

HCl

Auric chloride, treated with an excess of hydrochloric acid, forms hydrochloroauric acid.

$$AuCl_3 + HCl \rightarrow HAuCl_4$$

J. W. Mallet, Trans. Roy. Soc. (London), **180**, 406 (1889) *105*

$AuCl_3$

H_2 **III-1970**

Metallic gold is precipitated from its solutions by hydrogen gas.

$$(2AuCl_3 + 3H_2 \rightarrow 2Au + 6HCl)$$

G. Russell,
Ref., R. Gerstl, Ber., **6**, 1424 (1873) *11*

$AuCl_3$

H_2 **III-1971**

Hydrogen passed into gold chloride solution precipitates powdered gold.

$$2AuCl_3 + 3H_2 \rightarrow \underline{2Au} + (6HCl)$$

T. Svedberg, Ark. Kem., Min., Geol., **3**, 16, 5 (1909) *10*

$AuCl_3$

$H_2C_2O_4$ **III-1972**

Auric chloride was reduced with oxalic acid, forming gold, carbon dioxide, and hydrochloric acid.

$$2AuCl_3 + 3H_2C_2O_4 \rightarrow 2Au + (6CO_2 + 6HCl)$$

W. C. Roberts-Austin, Trans. Roy. Soc. (London), 349 (1888) *105*

$AuCl_3$

$H_2C_2O_4$ **III-1973**

A concentrated solution of oxalic acid precipitates gold from a solution of its chloride.

$$2AuCl_3 + 3H_2C_2O_4 \rightarrow \underline{2Au} + (6HCl + 6CO_2)$$

E. G. R. Ardagh, F. S. Seaborne and N. S. Grant, Can. Chem. & Met., **8**, 140 (1924) *15*

$AuCl_3$

H_2O_2 **III-1974**

Gold solutions in contact with hydrogen peroxide yield metallic gold and evolve oxygen.

$$2AuCl_3 + 3H_2O_2 \rightarrow \underline{2Au} + 3O_2 + (6HCl)$$

V. Lenher, J. Am. Chem. Soc., **36**, 1424 (1914) *1*

$AuCl_3$

III-1975

H_2S

A neutral aqueous solution of gold chloride is treated with hydrogen sulfide at room temperature. The precipitate is filtered ofi and washed with water. The precipitate is gold disulfide Au_2S_2.

$$8AuCl_3 + 9H_2S + 4H_2O \rightarrow \underline{4Au_2S_2} + 24HCl + H_2SO_4$$

G. Kruss, Ann., **238**, 36 (1887) *20*

$AuCl_3$

III-1976

H_2S

Gold chloride with hydrogen sulfide in water at a temp. of 90° will yield gold, hydrochloric acid and sulfuric acid.

$$8AuCl_3 + 3H_2S + 12H_2O \rightarrow 8Au + 24HCl + 3H_2SO_4$$

U. Antony and A. Lucchesi, Gazz. chim. ital., **20**, 601 (1890) *21*

$AuCl_3$

III-1977

H_2S

H_2O

Gold chloride with hydrogen sulfide in the presence of water and at a temp. of about 38° yields gold sulfide, hydrochloric acid and sulfuric acid.

$$8AuCl_3 + 9H_2S + 4H_2O \rightarrow 4Au_2S_2 + 24HCl + H_2SO_4$$

U. Antony and A. Lucchesi, Gazz. chim. ital., **20**, 601 (1890) *21*

$AuCl_3$

III-1978

HgCl

Gold chloride mixed with mercurous chloride in the presence of an excess of water and under ordinary conditions gives mercuric chloride and gold.

$$3HgCl + AuCl_3 \rightarrow 3HgCl_2 + Au$$

U. Antony and A. Lucchesi, Gazz. chim. ital., **26**[2], 196 (1896) *21*

$AuCl_3$

I_2 **III-1979**

Auric chloride solution is reduced to aurous iodide by free iodine.

$$AuCl_3 + 2I_2 \rightarrow AuI + 3ICl$$

F. Gramp, Ber., **7**, 1723 (1874) *11*

$AuCl_3$

KBr **III-1980**

Auric chloride, when treated with potassium bromide yields bromine.

$$AuCl_3 + 2KBr \rightarrow AuCl + 2KCl + Br_2$$

V. Lenher, J. Am. Chem. Soc., **35**, 734 (1913) *1*

$AuCl_3$

KCN **III-1981**

Solutions of auric chloride and potassium cyanide yield well-formed crystals of the double potassium auric cyanide and potassium chloride. Similar compounds may be made in which the potassium may be replaced by Na, NH_4, Ba, Cd, Zn, Sr, Ca or Co and the cyanogen of the auric cyanide may be replaced by Cl, Br or I.

$$AuCl_3 + 4KCN \rightarrow Au(CN)_3 \cdot KCN + 3KCl$$

C. G. Lindbom, Acta Univ. Lund., **12**, 2, No. 6, 12–43 (1876) *10*

$AuCl_3$

KI **III-1982**

Auric chloride and potassium iodide solutions react forming aurous iodide and free iodine.

$$AuCl_3 + 3KI \rightarrow AuI + I_2 + 3KCl$$

Victor E. Herschlag, Anal. Ed., **13**, 561 (1941) *44*

$AuCl_3$

KI **III-1983**

Auric chloride, when treated with potassium iodide liberates iodine.

$$AuCl_3 + 2KI \rightarrow AuCl + 2KCl + I_2$$

V. Lenher, J. Am. Chem. Soc., **35**, 734 (1913) *1*

$AuCl_3$

III-1984

$KMnO_4$

Potassium permanganate, on being allowed to stand for some time with a solution of auric chloride, causes metallic gold to be precipitated along with manganese dioxide.

$$AuCl_3 + 3KMnO_4 \rightarrow \underline{Au} + \underline{3MnO_2} + (3KCl + 3O_2)$$

V. Lenher, J. Am. Chem. Soc., **36**, 1424 (1914) *1*

$AuCl_3$

III-1985

$MgCO_3$

Magnesite reduces gold chloride to metallic gold above 310° under pressure.

$$2AuCl_3 + 3MgCO_3 \rightarrow 2Au + (3MgCl_2 + 1.5O_2 + 3CO_2)$$

H. H. Morris, J. Am. Chem. Soc., **40**, 927 (1918) *1*

$AuCl_3$

III-1986

$Mg(HCO_3)_2$

Gold hydroxide and metallic gold are formed when auric chloride is heated with magnesium bicarbonate at 322°.

$$3AuCl_3 + 3Mg(HCO_3)_2 \rightarrow Au + 2Au(OH)_3 + (3MgCl_2 + 6CO_2 + 3Cl)$$

H. H. Morris, J. Am. Chem. Soc., **40**, 927 (1918) *1*

$AuCl_3$

III-1987

MgO

Gold hydroxide is precipitated when a solution of auric chloride is treated with magnesium oxide.

$$2AuCl_3 + 3MgO + 3H_2O \rightarrow \underline{2Au(OH)_3} + (3MgCl_2)$$

H. H. Morris, J. Am. Chem. Soc., **40**, 920 (1918) *1*

$AuCl_3$

$Mn(C_2H_3O_2)_2$ **III-1988**

A neutral aqueous solution of gold trichloride is treated with an excess of an aqueous solution of manganese acetate. A precipitate of gold trioxide forms.

$$2AuCl_3 + 3Mn(C_2H_3O_2)_2 + 3H_2O \rightarrow \underline{Au_2O_3} + 3MnCl_2 + 6CH_3COOH$$

P. Schottländer, Ann., **217**, 320 (1883) *20*

$AuCl_3$

$Mn(C_2H_3O_2)_2$ **III-1989**

Br_2

Manganese dioxide, prepared by the action of bromine on manganese acetate precipitates metallic gold from slightly acid, neutral or alkaline solutions of gold.

(a) $Mn(C_2H_3O_2)_2 + Br_2 + 2H_2O \rightarrow \underline{MnO_2} + 2HC_2H_3O_2 + 2HBr$

(b) $3MnO_2 + 2AuCl_3 \rightarrow (3MnCl_2 + 3O_2) + \underline{2Au}$

V. Lenher, J. Am. Chem. Soc., **36**, 1424 (1914) *1*

$AuCl_3$

NH_4OH **III-1990**

A precipitate of fulminating gold was formed by mixing auric chloride and ammonium hydroxide.

(a) $AuCl_3 + 3NH_4OH \rightarrow Au(OH)_3 + (3NH_4Cl)$

(b) $2Au(OH)_3 \rightarrow Au_2O_3 + (3H_2O)$

(c) $Au_2O_3 + 4NH_4OH \rightarrow Au_2O_3 \cdot 4NH_3 + (4H_2O)$

J. W. Mallet, Trans. Roy. Soc. (London), **180**, 421 (1889) *105*

$AuCl_3$

III-1991

NH_4OH

KCN

Potassium auro-cyanide can be prepared for electrolysis by precipitating gold chloride with ammonium hydroxide and heating it with saturated potassium cyannide.

(a) $(2AuCl_3 + 6NH_4OH \rightarrow Au_2O + 6NH_4Cl + 3H_2O + O_2)$
(b) $(Au_2O + 4KCN + H_2O \rightarrow 2KAu(CN)_2 + 2KOH)$

J. W. Mallet, Am. Chem. J., **12**, 190 (1890) *17*

$AuCl_3$

III-1992

$NaBO_3$

Sodium perborate precipitates metallic gold from gold solutions.

$$2AuCl_3 + 6NaBO_3 + 9H_2O \rightarrow 2Au + (6NaCl + 6H_3BO_3 + 9O)$$

V. Lenher, J. Am. Chem. Soc., **36**, 1424 (1914) *1*

$AuCl_3$

III-1993

NaH_2PO_2

A green solution containing finely divided gold is formed when a solution of auric chloride is treated with a 10% solution of sodium hypophosphite in presence of a trace of sulfuric acid.

$$2AuCl_3 + 2NaH_2PO_2 + 3H_2O \rightarrow \underline{2Au} + (2NaCl + 2H_3PO_3 + 4HCl + O)$$

M. C. Lea, Am. J. Sci., **153**, 64
Ref., J. Am. Chem. Soc., **19**, 51 (1897) *1*

$AuCl_3$

III-1994

Na_2O_2

Sodium peroxide precipitates metallic gold from gold solutions.

$$2AuCl_3 + 3Na_2O_2 \rightarrow \underline{2Au} + 3O_2 + (6NaCl)$$

V. Lenher, J. Am. Chem. Soc., **36**, 1424 (1914) *1*

$AuCl_3$

$Na_2S_2O_3$ **III-1995**

Sodium aurothiosulfate is formed when an aqueous solution of auric chloride reacts with sodium thiosulfate dissolved in water.

$$AuCl_3 + 2Na_2S_2O_3 \rightarrow Na_3Au(S_2O_3)_2 + (NaCl + Cl_2)$$

M. J. Fordos and A. Gélis, Ann. chim. phys. [3] **13**, 394 (1845)
Ref., McCluskey and Eichelberger, J. Am. Chem. Soc., **48**, 137 (1926) *1*

$AuCl_3$

$Na_2S_2O_3$ **III-1996**

Aurous sodium thiosulfate is formed when a solution of sodium thiosulfate is precipitated with auric chloride solution.

$$8Na_2S_2O_3 + 2AuCl_3 \rightarrow \underline{2Na_3Au(S_2O_3)_2} + 2Na_2S_4O_6 + 6NaCl$$

M. J. Fordos and A. Gélis, Ann. chim. phys. [3], **13**, 394 (1845)
Ref., J. Am. Chem. Soc., **22**, 109 (1900) *1*

$AuCl_3$

$Na_2S_2O_4$ **III-1997**

Solutions of sodium hyposulfite reduce solutions of gold chloride to metallic gold.

$$2AuCl_3 + 3Na_2S_2O_4 \rightarrow 2Au + 6NaCl + 6SO_2$$

O. Brunck, Ann., **327**, 245 (1903) *20*

$AuCl_3$

Na_2Te **III-1998**

Sodium telluride reduces gold chloride solution to metallic gold.

$$2AuCl_3 + Na_2Te \rightarrow 2NaCl + TeCl_4 + \underline{2Au}$$

C. A. Tibbals, Jr., J. Am. Chem. Soc., **31**, 911 (1909) *1*

$AuCl_3$

III-1999

$Na_8Fe_2(P_2O_7)_3$

Sodium ferropyrophosphate precipitates gold from its solutions. In dilute solutions a blue or violet-blue colloidal solution is obtained; more concentrated solutions cause precipitation of the slightly impure metal.

$$2AuCl_3 + 3Na_8Fe_2(P_2O_7)_3 \rightarrow \underline{2Au} + (6NaCl + 3Na_6Fe_2(P_2O_7)_3)$$

P. Pascal, Compt. rend., **146**, 862 (1908) *88*

$AuCl_3$

III-2000

OsO_4

NaOH

Osmium tetroxide solution made alkaline with sodium hydroxide precipitates gold from gold solutions.

$$2AuCl_3 + 2OsO_4 + 10NaOH \rightarrow \underline{2Au} + (6NaCl + 5H_2O + 2Na_2OsO_4) + 2.5O_2$$

V. Lenher, J. Am. Chem. Soc., **36**, 1424 (1914) *1*

$AuCl_3$

III-2001

PbO_2

Lead peroxide precipitates gold from gold solutions which are either neutral or alkaline.

$$2AuCl_3 + 3PbO_2 \rightarrow \underline{2Au} + (3PbCl_2 + 3O_2)$$

V. Lenher, J. Am. Chem. Soc., **36**, 1424 (1914) *1*

$AuCl_3$

III-2002

Pb_3O_4

Red lead precipitates metallic gold from gold solutions which are either neutral or alkaline.

$$2AuCl_3 + Pb_3O_4 \rightarrow \underline{2Au} + (3PbCl_2 + 2O_2)$$

V. Lenher, J. Am. Chem. Soc., **36**, 1424 (1914) *1*

$AuCl_3$("Au_2Cl_3")

Pt **III-2003**

Platinum foil becomes covered with a coating of gold when warmed for 18 hours in a solution of auric chloride.

$$2AuCl_3 + 3Pt \rightarrow 3PtCl_2 + 2Au(\text{"}Au_2Cl_3\text{"})$$

C. Saintpierre, Compt. rend., **54**, 1079 (1862) *29*

$AuCl_3$

Zn **III-2004**

When auric chloride is treated with zinc, gold and zinc chloride are formed.

$$3Zn + 2AuCl_3 \rightarrow \underline{2Au} + 3ZnCl_2$$

J. W. Mallet, Trans. Roy. Soc. (London), **180**, 431 (1889) *105*

$AuCl_3$

Adenine **III-2005**

When auric chloride is reacted with solutions of adenine hydrochloride a yellow crystalline precipitate of adenine chloroaurate is formed. Decomposes at 278–280°C.

$$C_5H_5N_5 \cdot HCl + AuCl_3 \rightarrow \underline{C_5H_5N_5HCl \cdot AuCl_3}$$

Y. Hiwatari, J. Biochem. (Japan), **7**, 164 (1927) *61*

$AuCl_3$

Betaine **III-2006**

When betaine hydrochloride is reacted with auric chloride solutions, yellow insoluble prisms are formed. M.P. 235°.

$$C_5H_{11}NO_2 \cdot HCl + AuCl_3 \rightarrow \underline{C_5H_{11}NO_2 \cdot HCl \cdot AuCl_3}$$

Y. Hiwatari, J. Biochem. (Japan), **7**, 172 (1927) *61*

$AuCl_3$

III-2007

Choline

When choline chloride is reacted with solutions of auric chloride a yellow flaky precipitate is formed. M.P. 256–265°C.

$$C_5H_{14}NO_2Cl + AuCl_3 \rightarrow \underline{C_5H_{14}NO_2Cl \cdot AuCl_3}$$

Y. Hiwatari, J. Biochem. (Japan), **7**, 166 (1927) *61*

$AuCl_3$

III-2008

Cinnamyl

Ephedrine · HCl

A test solution of gold chloride when mixed with a solution of cinnamyl ephedrine hydrochloride forms a yellow precipitate consisting of oily or resinous material.

$$C_{19}H_{24}ONCl + AuCl_3 \rightarrow \underline{C_{19}H_{24}ON \cdot AuCl_4}$$

Welsh and Keenan, J. Am. Pharm. Assn., **30**, 123 (1941) *112*

$AuCl_3$

III-2009

p-phenylenediamine

H_2SO_4

A 0.5% water solution of auric chloride is treated with a H_2SO_4 m-phenylenediamine solution (5:1000) A yellow to dark brown develops due to the reduction of the gold salt to metallic gold in colloidal form. A 5% $AuCl_3$ solution gives a violet color.

$$AuCl_3 + C_6H_4(NH_2)_2 \rightarrow AuCl_2 \cdot C_6H_3(NH_2)_2 + HCl$$

J. A. Siemssen, Chem. Ztg., **36**, 934 *25*

$AuCl_3$

III-2010

N,N′ dimethyl putrescine

HCl

Auric chloride if dissolved in dilute hydrochloric acid acts upon N,N′ dimethyl putrescine under precipitation of the chloroaurate of this base.

$$(2AuCl_3 + 2HCl + C_6H_{16}N_2 \rightarrow \underline{C_6H_{16}N_2 \cdot 2HAuCl_4})$$

W. Keil, Z. physiol. Chem., **196**, 81 (1931) *14*

$AuCl_3$

Hypoxanthine **III-2011**

When hypoxanthine hydrochloride is reacted with solutions of auric chloride an orange yellow crystalline precipitate is formed. M.P. 245–248°.

$$C_5H_4N_4O \cdot HCl + AuCl_3 \rightarrow \underline{C_5H_5N_4OHCl \cdot AuCl_3}$$

Y. Hiwatari, J. Biochem. (Japan), **7**, 165 (1927) *61*

$AuCl_3$

Light **III-2012**

When in contact with organic substances auric chloride is changed by light to aurous chloride, and then to metallic gold.

(a) $$AuCl_3 \rightarrow AuCl + Cl_2$$

(b) $$2AuCl \rightarrow \underline{2Au} + Cl_2$$

F. P. Venable, J. Am. Chem. Soc., **21**, 223 (1899) *1*

$AuCl_3$

Putrescine **III-2013**

When putrescine hydrochloride is reacted with auric chloride solutions a yellow precipitate is formed. The prismatic crystals melt at 235–238°C.

$$C_4H_{12}N_2 \cdot 2HCl + 2AuCl_3 \rightarrow \underline{C_4H_{12}N_2 \cdot 2[HCl \cdot AuCl_3]}$$

Y. Hiwatari, J. Biochem. (Japan), **7**, 171 (1927) *61*

$AuCl_3$

Stachydrine **III-2014**

When stachydrine hydrochloride is reacted with solutions of auric chloride, a yellow precipitate of rhombic crystals is formed. M.P. 232°.

$$C_7H_{13}NO_2 \cdot HCl + AuCl_3 \rightarrow \underline{C_7H_{13}NO_2 \cdot HClAuCl_3}$$

Y. Hiwatari, J. Biochem. (Japan), **7**, 173 (1927) *61*

$AuCl_3$

III-2015

N,N,N′-trimethyl putrescine

HCl

Auric chloride dissolved in dilute hydrochloric acid acts upon N,N,N′ trimethyl putrescine under precipitation of the chloroaurate of this base.

$$(2AuCl_3 + 2HCl + C_7H_{18}N_2 \rightarrow \underline{C_7H_{18}N_2 \cdot 2HAuCl_4})$$

W. Keil, Z. physiol. Chem., **196**, 81 (1931) *14*

$AuCl_3$

III-2016

ε

From a very concentrated solution of gold chloride as neutral as possible, gold is obtained by passing an electric current of small voltage through it.

$$2AuCl_3 \rightarrow 2Au + 3Cl_2$$

M. Becquerel and E. Becquerel. Compt. rend., **55**, 19 (1862) *29*

$AuCl_3$

III-2017

ε

Gold chloride in a neutral solution is decomposed by an electrical current to precipitate gold in its pure state and to give off free chlorine.

$$(2AuCl_3 \rightarrow \underline{2Au} + 3Cl_2)$$

M. Becquerel, Chem. News, **6**, 126 (1862) *101*

$AuCl_3$

III-2018

Δ

Auric chloride was heated, giving aurous chloride.

$$AuCl_3 \rightarrow AuCl + (Cl_2)$$

J. W. Mallet, Trans. Roy. Soc. (London), **180**, 410 (1889) *105*

$AuCl_4^-$

H_2O **III-2019**

ε

The electrochemical behavior of gold in acid and neutral solutions is studied and the hydrolysis of the various complex gold anions is investigated.

(a) $AuCl_4^- + H_2O \rightleftharpoons AuCl_3OH^- + HCl$

(b) $AuCl_2^- + H_2O \rightleftharpoons AuClOH^- + HCl$

G. Grube, Z. Elektrochem., **35**, 705 (1929) *86*

$AuHCl_4$

Ag_2CO_3 **III-2020**

A solution of aurichloric acid is warmed and a suspension of silver carbonate is added in small portions. Carbon dioxide is evolved and a black precipitate of auric trioxide forms.

$$2AuHCl_4 + 4Ag_2CO_3 \rightarrow \underline{Au_2O_3} + 8AgCl + H_2O + 4CO_2$$

P. Schottländer, Ann., **217**, 313 (1883) *20*

$AuOHSO_4$

H_2O **III-2021**

Auric hydroxo sulfate is dissolved in concentrated sulfuric acid and then poured into water. Auric hydroxo oxide is produced.

$$AuOHSO_4 + H_2O \rightarrow AuOOH + H_2SO_4$$

P. Schottländer, Ann., **217**, 347 (1883) *20*

$AuOHSO_4$

$KHSO_4$ **III-2022**

One part of potassium hydrogen sulfate is dissolved in ten parts of auric hydroxosulfate in aqueous solution by gentle warming and the solution evaporated to dryness on a sand bath at 200°C.

$$AuOHSO_4 + KHSO_4 \rightarrow KAu(SO_4)_2 + H_2O$$

P. Schottländer, Ann., **217**, 373 (1883) *20*

$Au(OH)_2NH_2$

III-2023

KOH

If the above auriamine is boiled with potassium hydroxide, one-half of its nitrogen is lost as ammonia and there remains a dark brown, flocculent, fulminating product of greater explosive power than the original compound. This new product is thought to be a biauriamine.

$$2Au(OH)_2NH_2 \rightarrow Au_2(OH)_4NH + NH_3$$

J. Jacobsen, Compt. rend., **146**, 1214 (1908) *38*

$Au(OH)_3 \cdot 4AgCl$

III-2024

NH_3

If the precipitate formed by the action of silver nitrate on chlorauric acid is treated with ammonia, the silver chloride dissolves and a flocculent precipitate of an auriamine is obtained by the following reaction.

$$Au(OH)_3 + NH_3 \rightarrow \underline{Au(OH)_2NH_2} + H_2O$$

J. Jacobsen, Compt. rend., **146**, 1214 (1908) *38*

AuOOH

III-2025

HNO_3

Completely dried auric hydroxo oxide is treated with 3.6 parts of concentrated nitric acid which must be free from nitrous acid in a boiling water bath until a clear yellow solution is formed. The solution is cooled in an ice salt bath and crystals of the compound $HAu(NO_3)_4$ with three H_2O separate.

$$AuOOH + 4HNO_3 \rightarrow \underline{HAu(NO_3)_4} + 2H_2O$$

P. Schottländer, Ann., **217**, 359 (1883) *20*

$AuSO_4$

III-2026

H_2O

An aqueous solution of gold monosulfate is boiled and a precipitate of the composition shown below forms.

$$3AuSO_4 + 4H_2O \rightarrow \underline{Au_3O_2(OH)_2} + 3H_2SO_4$$

P. Schottländer, Ann., **217**, 337 (1883) *20*

$Au_2O_2(OH)_2 \cdot 2H_2O$

$Ba(OH)_2$ **III-2027**

$Ca(OH)_2$

$Sr(OH)_2$

Aurates of alkali earths are prepared by treating the hydroxide with auryl hydroxide.

(a) $Au_2O_2(OH)_2 \cdot 2H_2O + Ba(OH)_2 + 2H_2O \rightarrow Ba(AuO_2)_2 \cdot 6H_2O$

(b) $Au_2O_2(OH)_2 \cdot 2H_2O + Ca(OH)_2 + 2H_2O \rightarrow Ca(AuO_2)_2 \cdot 6H_2O$

(c) $Au_2O_2(OH)_2 \cdot 2H_2O + Sr(OH)_2 + 2H_2O \rightarrow Sr(AuO_2)_2 \cdot 6H_2O$

F. Meyer, Compt. rend., **145**, 805 (1907)
Ref., E. Horton, J. Chem. Soc. (London), **94**:2, 47 (1908) *57*

Au_2O_3

CO **III-2028**

Auric oxide is reduced at 0° by carbon monoxide.

$$Au_2O_3 + 3CO \rightarrow 2Au + 3CO_2$$

I. W. Fay and A. F. Seeker, J. Am. Chem. Soc., **25**, 647 (1903) *1*

Au_2O_3

HI **III-2029**

Hydriodic acid and auric oxide yield aurous iodide and iodine.

$$Au_2O_3 + 6HI \rightarrow 2AuI + 3H_2O + 2I_2$$

W. B. Pollard, J. Chem. Soc. (London), **2**, 1348 (1926) *65*

Au_2O_3

H_2O_2 **III-2030**

Hydrogen peroxide reduces auric oxide to metallic gold.

$$Au_2O_3 + 3H_2O_2 \rightarrow 2Au + 3H_2O + 3O_2$$

V. Lenher, J. Am. Chem. Soc., **36**, 1424 (1914) *1*

Au_2O_3

III-2031

KOH

Auric oxide is converted into potassium aurate when treated with an excess of potassium hydroxide.

$$Au_2O_3 + 2KOH \rightarrow 2KAuO_2 + (H_2O)$$

M. E. Fremy, Ann. chim. phys. [3], **31**, 481 (1851)
Ref. Roseveare and Buehrer, J. Am. Chem. Soc., **49**, 1222 (1927) *1*

Au_2O_3

III-2032

$Na_2S_2O_3$

To an aqueous solution of hydrochloroauric acid enough NaOH solution is added to precipitate gold hydroxide. This solution is then poured into a solution of sodium thiosulfate with stirring. Addition of acid (HNO_3) dissolves the gold hydroxide (a). Further addition of acid to neutrality discharges the red color (b). Treatment of the filtrate with alcohol precipitates white sodium aurothiosulfate.

(a) $Au_2O_3 + 4Na_2S_2O_3 + 2H_2O \rightarrow 4NaOH + 2Na_2S_4O_6 + Au_2O$

(b) $Au_2O + 4Na_2S_2O_3 + H_2O \rightarrow 2NaOH + \underline{2Na_3Au(S_2O_3)_2}$

H. Brown, J. Am. Chem. Soc., **49**, 959 (1927) *16*

$Au_2O_3 \cdot 4NH_3$

III-2033

KCN

Fulminating gold was heated with potassium cyanide to form potassium aurocyanide.

(a) $Au_2O_3 \cdot 4NH_3 + 2KCN \rightarrow (K_2O) + 2AuCN + (O_2 + 4NH_3)$

(b) $AuCN + KCN \rightarrow KAu(CN)_2$

J. W. Mallet, Trans. Roy. Soc. (London), **180**, 421 (1889) *105*

Au_2S_3

III-2034

KCN

Gold sulfide reacts with potassium cyanide to give gold potassium cyanide, potassium sulfide, and potassium thiocyanate.

$$Au_2S_3 + 6KCN \rightarrow 2KAu(CN)_2 + K_2S + 2KCNS$$

U. Antony and A. Lucchesi, Gazz. chim. ital., **21**[2], 210 (1891) *21*

$Au_3O_2(OH)_2$

HCl **III-2035**

Δ

Gold monooxyhydrate is decomposed by heating with concentrated hydrochloric acid to form metallic gold and gold trioxide.

$$Au_3O_2(OH)_2 + \Delta \rightarrow Au + Au_2O_3 + H_2O$$

P. Schottländer, Ann., **217**, 340 (1883) *20*

$(CH_3CH_2CH_2)_2AuCN$

Δ **III-2036**

A linear, polymeric structure is now suggested for dipropyldicyanodigold, obtained by heating dipropylmonocyanomonogold above 80°.

$$3(CH_3CH_2CH_2)_2AuCN \rightarrow (CH_3CH_2CH_2)_2Au_2(CN)_2 + AuCN + 2CH_3(CH_2)_4CH_3$$

C. S. Gibson et. al., J. Chem. Soc. (London), 1024 (1935)
Ref., C. S. Gibson, Proc. Roy. Soc. (London), **173A**, 160 (1939) *110*

$(CH_3)_3NHAuCl_4$

Δ **III-2037**

Trimethylamine chloroaurate, when heated, will form metallic gold.

(a) $(CH_3)_3NHAuCl_4 \rightarrow (AuCl_3 + HCl + (CH_3)_3N)$

(b) $2AuCl_3 \rightarrow 2Au + (3Cl_2)$

J. W. Mallet, Trans. Roy. Soc. (London), **180**, 414 (1889) *105*

$CsAuCl_4$

$Na_2S_2O_3$ **III-2038**

To a solution of sodium thiosulfate, cesium auric chloride (from gold dissolved in aqua regia and precipitated by cesium nitrate) is added in small quantities with stirring. Addition of 95% alcohol precipitates white crystalline sodium aurothiosulfate.

$$CsAuCl_4 + 4Na_2S_2O_3 \rightarrow \underline{Na_3Au(S_2O_3)_2} + Na_2S_4O_6 + 3NaCl + CsCl$$

K. L. McCluskey and L. Eichelberger, J. Am. Chem. Soc., **48**, 136 (1928) *16*

$HAuBr_4$

III-2039

$HCHO_2$

Formic acid was used to reduce hydrobromoauric acid to metallic gold.

$$3HCHO_2 + 2HAuBr_4 \rightarrow 2Au + (8HBr + 3CO_2)$$

J. W. Mallet, Trans. Roy. Soc. (London), **180**, 406 (1889) *105*

$HAuBr_4$

III-2040

$H_2C_2O_4$

Oxalic acid was used to reduce hydrobromoauric acid to metallic gold.

$$3H_2C_2O_4 + 2HAuBr_4 \rightarrow 2Au + (8HBr + 6CO_2)$$

J. W. Mallet, Trans. Roy Soc. (London), **180**, 406 (1889) *105*

$HAuCl_4$

III-2041

$AgNO_3$

Auric chloride solution with excess silver nitrate yields a dark yellow-red precipitate, which is shown to be a mixture of gold oxide and silver chloride.

$$2HAuCl_4 + 8AgNO_3 + 3H_2O \rightarrow \underline{Au_2O_3} + \underline{8AgCl} + 8HNO_3$$

E. Wohlwill, Z. Elektrochem. **4**, 382 (1898) *86*

$HAuCl_4$

III-2042

$AgNO_3$

A series of reactions take place when chlorauric acid, dissolved in ice water, is treated with a cold solution of silver nitrate.

(a) $$HAuCl_4 + AgNO_3 \rightarrow \underline{AgAuCl_4} + HNO_3$$

(b) $$HAuCl_4 + 4AgNO_3 \rightarrow \underline{4AgCl} + Au(NO_3)_3 + HNO_3$$

(c) $$HAuCl_4 + AgNO_3 \rightarrow AuCl_3 + \underline{AgCl} + HNO_3$$

Lengfeld, Am. Chem. J., **26**, 330 (1901) *1*

$HAuCl_4$

Ag_2CO_3 **III-2043**

A mixture of auric oxide and silver chloride is formed when chlorauric acid is treated with silver carbonate.

$$2HAuCl_4 + 4Ag_2CO_3 \rightarrow 8AgCl + Au_2O_3 + H_2O + 4CO_2$$

P. Schottländer, Ann., **217**, 312 (1883)
Ref., Lengfeld, Am. Chem. J., **26**, 330 (1901) *1*

$HAuCl_4$

$C_6H_6O_2$ **III-2044**

Auric salts when treated with hydroquinone in a solution buffered with potassium bifluoride give a precipitate of the metal.

$$2HAuCl_4 + 3C_6H_6O_2 \rightarrow \underline{2Au} + (3C_6H_4O_2 + 8HCl)$$

W. B. Pollard, Analyst, **62**, 597 (1937) *40*

$HAuCl_4$

KOH **III-2045**

Auric oxide precipitates when chlorauric acid is treated with potassium hydroxide.

$$2HAuCl_4 + 3KOH \rightarrow \underline{Au_2O_3} + (3KCl + 5HCl)$$

M. E. Fremy, Ann. chim. phys. [3], **31**, 481 (1851)
Ref., Roseveare and Buehrer, J. Am. Chem. Soc., **49**, 1221 (1927) *1*

$HAuCl_4$

2-Aminoheptane **III-2046**

2-Aminoheptane combines with chlorauric acid yielding a chloraurate melting at 77 to 78.5°C.

$$C_5H_{11}(CH_3)CHNH_2 + HAuCl_4 \rightarrow C_5H_{11}CHNH_2 \cdot CH_3 \cdot HAuCl_4$$

Dunker, Hartung and Chapman, J. Am. Pharm. Assn., **30**, 623 (1941) *112*

$HAuCl_4 \cdot 3H_2O$

III-2047

$AgNO_3$

When silver nitrate is added to a solution of chlorauric acid, a light brown precipitate is obtained which corresponds to the formula given below.

$$HAuCl_4 \cdot 3H_2O + 4AgNO_3 \rightarrow \underline{Au(OH)_3 \cdot 4AgCl} + 4HNO_3$$

J. Jacobsen, Compt. rend., **146**, 1213 (1908) *38*

$HAu(NO_3)_4$

III-2048

H_2SO_4

Dry auric hydrogen nitrate is heated with five parts of 80% sulfuric acid at 100°C until all nitric acid is decomposed; then the heat is slowly raised to 200°C. Upon cooling, hydroxyauric sulfate separates from the solution.

$$HAu(NO_3)_4 + H_2SO_4 \rightarrow \underline{AuOHSO_4} + 4NO_2 + H_2O + O_2$$

P. Schottländer, Ann., **217**, 368 (1883) *20*

$KAuBr_4$

III-2049

H_2

Δ

Gold can be reduced from potassium auri-bromide, by heating the dry salt in a stream of hydrogen.

$$(2KAuBr_4 + 3H_2 \rightarrow 2KBr + 6HBr + 2Au)$$

G. Kruss, Ann., **238**, 30 (1886)
Ref., J. W. Mallet, Am. Chem. J., **12**, 77 (1890) *17*

$KAuBr_4$

III-2050

H_2SO_3

Potassium auribromide in solution can be reduced to metallic gold by sulfurous acid.

$$(2KAuBr_4 + H_2SO_3 \rightarrow 2HBr + K_2SO_3 + 3Br_2 + 2Au)$$

G. Kruss, Ann., J., **238**, 30 (1886)
Ref., J. W. Mallet, Am. Chem. J., **12**, 77 (1890) *17*

$KAuBr_4$

H_2SO_3 **III-2051**

KOH

Potassium auricbromide is dissolved in water and treated with sulfurous acid. The compound is reduced to potassium aurobromide. Treatment of the potassium aurobromide with dilute potassium hydroxide forms aurous oxide.

(a) $KAuBr_4 + H_2SO_3 + H_2O \rightarrow KAuBr_2 + 2HBr + H_2SO_4$

(b) $2KAuBr_2 + 2KOH \rightarrow Au_2O + 4KBr$

G. Kruss, Ann., **237**, 275 (1887) *20*

$KAuBr_4$

Δ **III-2052**

Potassium auribromide, when heated with a bunsen flame, will break down leaving metallic gold.

$$(2KAuBr_4 \rightarrow 2Au + 2KBr + 3Br_2)$$

Thorpe and Laurie, J. Chem. Soc. (London), Dec. 1887, p. 866
Ref., J. W. Mallet, Am. Chem. J., **12**, 79 (1890), *17*

$KAu(CN)_2$

H_2O **III-2053**

ε

Metallic gold can be obtained by subjecting potassium aurocyanide to electrolysis.

$$(4KAu(CN)_2 + 2H_2O \rightarrow 4KCN + 4HCN + 4Au + O_2)$$

J. W. Mallet, Am. Chem. J., **12**, 195 (1890) *17*

$KAu(CN)_2$

III-2054

Zn

KCN

Zinc dust precipitates gold from its solution in potassium cyanide. The reaction is greatly catalyzed by the addition of lead acetate which causes the zinc to precipitate metallic lead according to the formula, which speeds up the action.

$$2KAu(CN)_2 + 4KCN + 2Zn + 2H_2O \rightarrow \underline{2Au} + 2K_2Zn(CN)_4 + 2KOH + H_2$$

C. Spearman, Can. Chem. & Met., **9**, 56 (1925) *15*

$KAuCl_4$

III-2055

Ag_2CO_3

Silver chloride is formed when a solution of anhydrous potassium chloraurate in absolute alcohol is shaken with silver carbonate.

$$2KAuCl_4 + Ag_2CO_3 \rightarrow K_2CO_3 + \underline{2AgCl} + 2AuCl_3$$

F. Lengfeld, Am. Chem. J., **26**, 331 (1901) *1*

$KAuO_2$

III-2056

H_2SO_4

Auric oxide is precipitated when potassium aurate is treated with sulfuric acid.

$$2KAuO_2 + H_2SO_4 \rightarrow \underline{Au_2O_3} + (K_2SO_4 + H_2O)$$

M. E. Fremy, Ann. chim. phys. [3], **31**, 481 (1851)
Ref., Roseveare and Buehrer, J. Am. Chem. Soc., **49**, 1222 (1927) *1*

$NaAuCl_2$

$Na_2S_2O_3$ **III-2057**

A few cc. of NaCl solution were added to 2 cc. of a gold chloride solution (0.0382 g. of gold per cc.) and sulfurous acid run in from a buret until a colorless solution was obtained. This solution contains sodium aurous chloride. The colorless solution was added to a sodium thiosulfate solution in which barium carbonate was suspended. After filtration, sodium aurothiosulfate is precipitated by 95% alcohol.

$$NaAuCl_2 + 2Na_2S_2O_3 \rightarrow Na_3Au(S_2O_3)_2 + 2NaCl$$

K. L. McCluskey and L. Eichelberger, J. Am. Chem. Soc., **48**, 136 (1926) *16*

$Na_3Au(S_2O_3)_2$

C_2H_2 **III-2058**

A yellow flocculent precipitate of aurous carbide forms when a slow stream of acetylene gas is passed through a strongly ammoniacal solution of aurous sodium thiosulfate.

$$2Na_3Au(S_2O_3)_2 + C_2H_2 \rightarrow \underline{Au_2C_2} + (3Na_2S_2O_3 + H_2S_2O_3)$$

J. A. Mathews and L. L. Watters, J. Am. Chem. Soc., **22**, 109 (1900) *1*

$HfCl_4$

III-2059

Sodium Oleate

Hafnium soaps may be prepared by reaction of a salt of a fatty acid with a hafnium salt.
Optimum temp. is 40–70°C.
Oleic salt reaction is shown as an example.

$$4CH_3(CH_2)_7HC{:}CH(CH_2)_7COONa + HfCl_4 \rightarrow [CH_3(CH_2)_7HC{:}CH(CH_2)_7COO]_4Hf + 4NaCl$$

Brit. Pat. 395, 406, July, 17 1933
Ref., Oil and Soap, **11**, 4, 77 (1934) *111*

HfI_4

III-2060

Δ

Pure hafnium can be obtained by heating the tetraiodide in a pyrex bulb at 600°C, the bulb containing a tungsten filament at 2000°C in contact with which the hafnium tetraiodide dissociates depositing the metal.

$$HfI_4 \rightarrow Hf + 2I_2$$

Can. Chem. & Met., **15**, 150 (1931) *15*

$HfOCl_2$

III-2061

Na_2HPO_4

HCl

Hafnium pyrophosphate precipitates when hydrochloric acid solutions of hafnium oxychloride and disodium phosphate react.

$$HfOCl_2 + 2Na_2HPO_4 + 2HCl \rightarrow \underline{HfP_2O_7 \cdot 2H_2O} + (4NaCl)$$

G. Hevesy and K. Kimura, J. Am. Chem. Soc., **47**, 2541 (1925) *1*

$Ho_2(C_2O_4)_3$

Δ **III-2062**

When holmium oxalate is heated, holmium oxide is left.

$$Ho_2(C_2O_4)_3 + \Delta \rightarrow Ho_2O_3 + (3CO_2 + 3CO)$$

O. Holmberg, Ark. Kem., Min., Geol., **4**, Pt. 2, 3 (1910) *10*

C_2D_2

III-2063

DBr

Deutero ethylene can be prepared from deutero acetylene by the following reactions:

(a) $$C_2D_2 + 2DBr \rightarrow C_2D_4Br_2$$

(b) $$C_2D_4Br_2 + Zn \rightarrow C_2D_4 + ZnBr_2$$

H. S. Taylor, J. Chim. phys., **34**, 529 (1937) *69*

D

III-2064

CH_4

Deuterium atoms react with methane at about 500°C according to the equation:

$$D + CH_4 \rightarrow CH_3D + H$$

E. W. R. Steacie, Can. J. Res., **B15**, 264 (1937) *69*

D

III-2065

CH_4

The exchange reaction of deuterium atoms with methane has an activation energy of 11 Cals.

$$D + CH_4 \rightarrow CH_3D + H$$

K. H. Geib and E. W. R. Steacie, Trans. Roy. Soc. (Canada), [III] **29**, 91, (1935) *69*

D

III-2066

CH_4

NH_3

The mercury photosensitized exchange reactions of deuterium with methane, water and ammonia, and the decomposition of water and ammonia, have been investigated at temperatures of 30–600° and total pressures of 100 mm. With methane and ammonia, the exchange above 300° proceeds by the mechanism.

(a) $$D + CH_3H \rightarrow CH_3D + H$$

(b) $$D + NH_2H \rightarrow NH_2D + H$$

(c) $$H + D_2 \rightarrow HD + D$$

A. Farkas and H. W. Melville, Proc. Roy. Soc. (London), **157A**, 625 (1936)
H. W. Melville, ibid., **157A.**, 621 (1936) *110*

D

C_2H_2 **III-2067**

The exchange reaction of deuterium atoms with acetylene takes place through formation of a quasimolecule, C_2H_2D. The energy of activation is less than 5 Cals.

$$C_2H_2 + D \rightarrow C_2HD + H$$

K. H. Geib and E. W. R. Steacie, Trans. Roy. Soc. (Canada) [III], **29**, 91 (1935) *69*

D

NH_3 **III-2068**

The exchange reaction of deuterium atoms with ammonia has an activation energy of 11 Cals.

$$D + NH_3 \rightarrow NH_2D + H$$

K. H. Geib and E. W. R. Steacie, Trans. Roy. Soc., (Canada) [III] **29**, 91 (1935) *69*

D_2

AgCl **III-2069**

Deuterium is passed over silver chloride at 700° to give deuterium chloride. The deuterium chloride is frozen out and the deuterium is recycled.

$$D_2 + 2AgCl \rightarrow 2DCl + 2Ag$$

H. Steiner and E. K. Rideal, Proc. Roy. Soc. (London), **173A**, 504 (1939) *110*

D_2

CH_4 **III-2070**

Deuterium will react with methane at 1000°C to exchange a deuterium atom for a hydrogen atom.

(a) $$D_2 \rightarrow 2D$$

(b) $$D + CH_4 \rightarrow CH_3D + H$$

Farkas, J. Chem. Soc. (London), 26–35 (1936)
Ref., Steacie, Can. J. Res., **15B**, 265 (1937) *94*

D_2

CO_2 **III-2071**

Tetradeuteromethane can be prepared by a catalytic process in the gas phase. Deuterium and carbon dioxide react in the presence of a supported nickel catalyst at a temperature of 350°C.

$$CO_2 + 4D_2 \rightarrow CD_4 + 2D_2O$$

George E. MacWood and Harold C. Urey, J. Chem. Phy., **4**, 402 (1935) *44*

D_2

C_2H_6 **III-2072**

The reaction between ethane and deuterium proceeds after (a) between 90° and 140°, and according to (b) at 200°:

(a) $$C_2H_6 + D_2 \rightarrow C_2H_5D + HD$$

(b) $$C_2H_6 + D_2 \rightarrow 2CH_3D$$

H. S. Taylor, J. Chim. phys., **34**, 529 (1937) *69*

D_2

Cl **III-2073**

Light

The photochemical reaction of deuterium and chlorine (a) at ordinary temperatures proceeds more slowly than the corresponding reaction involving hydrogen (b).

(a) $$Cl + D_2 \rightarrow DCl + D$$

(b) $$Cl + H_2 \rightarrow HCl + H$$

H. S. Taylor, J. Franklin Inst., **218**, 22 (1934) *16*

D_2

HBr **III-2074**

Deuterium and hydrogen bromide form an equilibrium mixture containing deuterium bromide. Para hydrogen and hydrogen bromide give another equilibrium mixture.

$$D_2 + 2HBr \rightleftarrows H_2 + 2DBr$$

H. Steiner, Proc. Roy. Soc. (London), **173A**, 531 (1939) *110*

D_2

HCl **III-2075**

Deuterium and hydrogen chloride form an equilibrium mixture containing deuterium chloride. Equilibrium is also established between hydrogen and deuterium chloride. Para hydrogen and hydrogen chloride give another equilibrium mixture.

$$D_2 + 2HCl \rightleftarrows H_2 + 2DCl$$

H. Steiner and E. K. Rideal. Proc. Roy. Soc. (London), **173A**, 504 (1939) *110*

D_2

NH_3 **III-2076**

The exchange between the hydrogen of ammonia and deuterium proceeds at ordinary temperature with an iron catalyst:

$$NH_3 + D_2 \rightarrow NH_2D + HD$$

H. S. Taylor, J. Chim. phys., **34**, 529 (1937) *69*

D_2O

CH_3MgI **III-2077**

A solution of deuterium oxide in dioxane is added to methyl magnesium iodide to produce monodeuteromethane.

$$D_2O + CH_3MgI \rightarrow CH_3D + Mg(OD)I$$

W. H. J. Childs and H. A. Jahn, Proc. Roy. Soc. (London), **169A**, 428 (1939) *110*

D_2O

CH_3NO_2 **III-2078**

An exchange reaction involving approximately equal weights of D_2O and CH_3NO_2 can be used in the preparation of trideuteronitromethane.

$$3D_2O + 2CH_3NO_2 \rightleftarrows 2CD_3NO_2 + 3H_2O$$

Thomas P. Wilson, J. Chem. Phy., **11**, 361 (1943) *44*

D_2O

$(COOH)_2$ **III-2079**

Anhydrous oxalic acid is evaporated twice in vacuo with deuterium oxide to give crystals of $(COOD)_2 \cdot 2D_2O$, m. 95.5–97.5° compared to 99.8–100.7°, for the corresponding hydrogen compound.

$$(COOH)_2 + 3D_2O \rightarrow (COOD)_2 \cdot 2D_2O + H_2O$$

J. M. Robertson and A. R. Ubbelohde, Proc. Roy. Soc. (London), **170A**, 224 (1939) *110*

D_2O

C_6H_5COCl **III-2080**

Deuterium chloride may be prepared conveniently by the action of benzoyl chloride on deuterium oxide at elevated temperatures.

(a) $$D_2O + C_6H_5COCl \rightarrow C_6H_5COOD + DCl$$

(b) $$C_6H_5COCl + C_6H_5COOD \rightarrow (C_6H_5CO)_2O + DCl$$

H. C. Brown and C. Groot, J. Am. Chem. Soc., **64**, 2223 (1942) *16*

D_2O

H **III-2081**

The activation energy of the exchange reaction of hydrogen atoms with heavy water (a) is the same as that of deuterium atoms with water (b).

(a) $$H + D_2O \rightarrow HDO + D$$

(b) $$D + H_2O \rightarrow HDO + H$$

K. H. Geib, and E. W. R. Steacie, Trans. Roy. Soc. (Canada), [III], **29**, 91 (1935) *69*

D_2O

H_2O_2 **III-2082**

The following exchange reactions take place readily when heavy water is mixed with hydrogen peroxide:

(a) $$H_2O_2 + D_2O \rightarrow H_2O + D_2O_2$$

(b) $$H_2O_2 + D_2O_2 \rightarrow 2HDO_2$$

(c) $$H_2O + D_2O \rightarrow 2HDO$$

H. Erlenmeyer and H. Gärtner, Helv. Chim., **17**, 190 (1934)
Ref., P. A. Giguère and O. Maass, Can. J. Res., **B18**, 84 (1940) *69*

D_2O

KHF_2 **III-2083**

KH_2PO_4

$NaHSO_4$

$SrCl_2$

Repeated recrystallization of potassium hydrogen fluoride, potassium di-hydrogen phosphate, or sodium hydrogen sulfate from deuterium oxide yields the corresponding deutero compounds. Strontium chloride unites directly to form a hexadeuterate.

(a) $$2KHF_2 + D_2O \rightarrow 2KDF_2 + H_2O$$

(b) $$KH_2PO_4 + D_2O \rightarrow KD_2PO_4 + H_2O$$

(c) $$2NaHSO_4 + D_2O \rightarrow 2NaDSO_4 + H_2O$$

(d) $$SrCl_2 + 6D_2O \rightarrow SrCl_2 \cdot 6D_2O$$

A. R. Ubbelohde, Proc. Roy. Soc. (London), **173A**, 418 (1939) *110*

D_2O

Mg **III-2084**

In the investigation of the heat of formation of deuterium oxide an obvious method, analagous to that of H_2O (a), was the reaction of deuterium oxide with a metal to form the metallic oxide and deuterium. (b).

(a) $$H_2O + Mg \rightarrow H_2 + MgO$$

(b) $$D_2O + Mg \rightarrow D_2 + MgO$$

J. W. Knowlton and F. D. Rossini, B. S. J. Res. **19**, 607, 608, (1937) *16*

D_2O

$SOCl_2$ **III-2085**

Deuterium oxide reacts with thionyl chloride to yield sulfur dioxide and deuterium chloride.

$$D_2O + SOCl_2 \rightarrow SO_2 + 2DCl$$

H. C. Brown and C. Groot, J. Am. Chem. Soc., **64**, 223 (1942) *16*

D_2O

III-2086

ε

Deuterium oxide is passed through a discharge tube operated at 0.5 amperes and 1000 volts to obtain spectroscopic quantities of OD.

$$D_2O \rightarrow D + OD$$

M. Ishaq, Proc. Roy. Soc. (London), **159A**, 110 (1937) *110*

D_2O_2

(Au) **III-2087**

The catalytic decomposition of deuterium peroxide on metallic gold is much slower than that of hydrogen peroxide:

$$2D_2O_2 \rightarrow 2D_2O + O_2$$

P. A. Giguère and O. Maass, Can. J. Res., **B18**, 84 (1940) *69*

D_2O_2

HI **III-2088**

The reaction of deuterium oxide with hydrogen iodide proceeds 0.60 times as fast as the corresponding reaction in hydrogen peroxide.

(a) $D_2O_2 + HI \rightarrow D_2O + HIO$

(b) $H_2O_2 + HI \rightarrow H_2O + HIO$

Tsing-Lien Chang, Science, **100**, 29 (1944) *44*

D_2O_2

Decomp. **III-2089**

The decomposition of alkaline deuterium peroxide on the surface of glass is a first order reaction. The rate of the reaction is much smaller than that for hydrogen peroxide:

$$2D_2O_2 \rightarrow 2D_2O + O_2$$

P. A. Giguère and O. Maass, Can. J. Res., **B18**, 84 (1940) *69*

HD

H_2O **III-2090**

The exchange of deuterium gas with water is discussed.

$$HD + H_2O \rightarrow H_2 + HDO$$

J. A. V. Butler, Z. Elektrochem., **44**, 58 (1938) *86*

D_2O

H_2O **III-2091**

The heat of reaction between light and heavy water is discussed.

$$H_2O + D_2O \rightarrow 2HDO$$

E. Lange, Z. Elektrochem., **44**, 42 (1938) *86*

HOD

CH_3COCH_3 **III-2092**

The equilibrium constant of the exchange reaction between heavy water and acetone is about 2, i.e., the heavy hydrogen is preferentially present in the acetone.

$$CH_3COCH_3 + HOD \rightleftharpoons CH_3COCH_2D + H_2O$$

A. Farkas and L. Farkas, J. Chem. Phy., **2**, 468 (1934) *44*

AsH_3

I_2 **III-2093**

NaOH

An iodometric acidimetric method for the determination of arsenic is based on the following reactions.

(a) $$AsH_3 + 4I_2 + 4H_2O \rightarrow H_3AsO_4 + 8HI$$

(b) $$H_3AsO_4 + 8HI + 10NaOH \rightarrow Na_2HAsO_4 + 8NaI + 10H_2O$$

R. C. Wiley, J. P. Bewley and R. Irey, Anal. Ed., **4**, 396 (1932) *44*

$CH_3H_2PO_2$

III-2094

$BaCO_3$

When an aqueous solution of monomethylphosphinic acid is boiled with barium carbonate the acid salt is formed.

$$2CH_3H_2PO_2 + BaCO_3 \rightarrow (CH_3)_2H_2BaP_2O_6 + CO_2 + H_2O$$

A. W. Hofmann, Ber., **5**, 108 (1872) *11*

$CH_3H_2PO_2$

III-2095

NH_4OH

$AgNO_3$

The normal silver salt of monomethylphosphinic acid is formed when the acid is almost neutralized with ammonium hydroxide and then treated with silver nitrate.

(a) $CH_3H_2PO_2 + 2NH_4OH \rightarrow CH_3(NH_4)_2PO_2 + 2H_2O$

(b) $CH_3(NH_4)_2PO_2 + 2AgNO_3 \rightarrow CH_3Ag_2PO_2 + 2NH_4NO_3$

A. W. Hofmann, Ber., **5**, 107 (1872) *11*

$CH_3H_2PO_2$

III-2096

PbO

An aqueous solution of monomethyl-phosphinic acid when heated with lead oxide forms both the normal and acid lead salt.

(a) $CH_3H_2PO_2 + PbO \rightarrow CH_3PbPO_2 + H_2O$

(b) $2CH_3H_2PO_2 + PbO \rightarrow (CH_3)_2PbH_2P_2O_6 + H_2O$

A. W. Hofmann, Ber., **5**, 107 (1872) *11*

$(CH_3)_2HPO_2$

III-2097

Ag_2O

The silver salt of dimethylphosphinic acid is formed when a solution of dimethyl phosphinic acid is neutralized with silver oxide.

$$2(CH_3)_2HPO_2 + Ag_2O \rightarrow 2(CH_3)_2AgPO_2 + H_2O$$

A. W. Hofmann, Ber., **5**, 109 (1872) *11*

$(CH_3)_2HPO_2$

$BaCO_3$ **III-2098**

When dimethylphosphinic acid is heated with an excess of barium carbonate, the barium salt of the acid is formed.

$$2(CH_3)_2HPO_2 + BaCO_3 \rightarrow H_2O + CO_2 + (CH_3)_4 \cdot BaP_2O_4$$

A. W. Hofmann, Ber, **5**, 109 (1872) *11*

$(CH_3)_2HPO_2$

PbO **III-2099**

Lead oxide reacts with dimethylphosphinic acid to form the lead salt.

$$2(CH_3)_2HPO_2 + PbO \rightarrow (CH_3)_4PbP_2O_4 + H_2O$$

A. W. Hofmann, Ber., **5**, 109 (1872) *11*

CH_4

CO_2 **III-2100**

CH_4

The equilibria of the Armstrong-Hilditch reaction are investigated between 500 and 800°C.

$$CH_4 + 3CO_2 \rightleftarrows 4CO + 2H_2O$$

J. Schmidt and B. Neumann, Z. Elecktrochem., **38**, 925 (1932) *86*

$C_2H_5H_2PO_2$

Ag_2O **III-2101**

Silver oxide reacts with ethyl phosphinic acid to form the normal silver salt.

$$C_2H_5H_2PO_2 + Ag_2O \rightarrow C_2H_5Ag_2PO_2 + H_2O$$

A. W. Hofmann, Ber., **5**, 110 (1872) *11*

C_2H_5OH

III-2102

Linolenic acid

An unusual system of hydrogenation is that of heating oils with alcohols. 150 parts of sunflower oil with 19 parts ethyl alcohol, 0.4 to 2% suitable catalyst were autoclaved at 240–360°C; and 28–41 atm., for 15 min. The linolenic content was reduced and the oleic was increased. Best results were had with Cu-Ni catalyst.

Reaction shown as of free acids.

$$CH_3(CH_2HC{:}CH)_3(CH_2)_7COOH + 2CH_3CH_2OH \rightarrow CH_3(CH_2)_7HC{:}CH(CH_2)_7COOH + 2CH_3CHO$$

M. F. Belyakov and G. A. Ivanova, Masloboino Zhirovoe Delo, **12**, 537–539, (1936)

Ref., Review of Sci. Lit. on Fats and Oils, 1937, in Oil and Soap, **15**, 4, 95 (1938) *111*

$(C_2H_5)_2HPO_2$

III-2103

Ag_2O

A solution of diethylphosphinic acid when boiled with silver oxide forms the silver salt of the acid.

$$2(C_2H_5)_2HPO_2 + Ag_2O \rightarrow 2(C_2H_5)_2AgPO_2 + H_2O$$

A. W. Hofmann, Ber., **5**, 111 (1872) *11*

$C_2S_3N_2H_2$

III-2104

HI

Perthio cyanic acid $(HCNS)_2S$ reacts with nascent hydrogen iodide to form thiourea, hydrogen iodide, and carbon disulfide.

$$(HCNS)_2S + 2(H) + HI \rightarrow CS(NH_2)_2 + HI + CS_2$$

L. Glutz, Ber., **3**, 343 (1870) *11*

CaH_2

$SiCl_4$ **III-2105**

Silicon tetrachloride vapors passed over powdered calcium hydride at a temperature below red heat form hydrogen, hydrochloric acid and a small quantity of silicochloroform, along with calcium chloride and calcium silicide or amorphous silicon, depending on the reaction temperature. At low temperatures with excess calcium hydride, calcium silicide is formed (a); at high temperatures with excess silicon tetrachloride, amorphous silicon (b).

(a) $3SiCl_4 + 5CaH_2 \rightarrow SiHCl_3 + 4H_2 + HCl + CaSi_2 + 4CaCl_2$

(b) $2SiCl_4 + 2CaH_2 \rightarrow SiHCl_3 + H_2 + HCl + 2CaCl_2 + Si$

A. Besson and L. Fournier, Compt. rend., **148**, 556–557 (1909) *38*

H

HNO_3 **III-2106**

Nascent hydrogen produced either electrolytically or by adsorption on platinum reduces nitric acid, liberating heat.

(a) $HNO_3 + 2H \rightarrow HNO_2 + (H_2O)$

(b) $HNO_3 + H \rightarrow NO_2 + (H_2O)$

(c) $HNO_3 + 8H \rightarrow NH_3 + (3H_2O)$

J. H. Gladstone and A. J. Tribe, J. Chem. Soc. (London), **35**, 172 (1879) *91*

H

H_2SO_4 **III-2107**

Hydrogen adsorbed on platinum or palladium reduces sulfuric acid forming sulfur dioxide. The reaction is more energetic in the case of palladium.

$$H_2SO_4 + 2H \rightarrow 2H_2O + SO_2$$

J. H. Gladstone and A. J. Tribe, J. Chem. Soc. (London), **35**, 172 (1879) *91*

H

III-2108

N_2

One part hydrogen and 78 parts air charged with water vapor passed over reduced iron wire at a rate of 100 l. air per 4½ hrs. form, at the optimum temp. of 300–350°, 204 mg. ammonia. Better results were obtained with dry peat, 441.2 g. peat yielding 11 g. ammonia, 2.75 g. of which represent synthetic ammonia.

$$3H_2 + \underset{\text{(air)}}{N_2} \rightarrow 2NH_3$$

Woltereck, Compt. rend., **146**, 124–5 (1908) *38*

(H)

III-2109

Bac

O_2

Pneumococcal and streptococcal cultures, and a few other coccal forms, which have abundant access to oxygen, form hydrogen peroxide.

$$2(H) + O_2 \rightarrow H_2O_2$$

J. W. McLeod and J. Gordon, Biochem. J., **16**, 499 (1922) *53*

H

III-2110

Cystine

When an acid solution of cystine is treated with a small amount of powdered zinc, the cystine is reduced to cysteine.

$$C_6H_{12}O_4N_2S_2 + 2H \rightarrow 2C_3H_6O_2NSH$$

L. Okabi, J. Biochem. (Japan), **8**, 411 (1928) *61*

H^+

III-2111

H_2O

Hydrogen ion is strongly solvated, i.e. it combines with water molecules. This fact explains the decrease of the catalytic effect of the hydrogen ion in alcoholic solutions as soon as water is added.

$$H^+ + H_2O \rightarrow (H_3O)^+$$

Samuel H. C. Briggs, J. Chem. Soc. (London), **115:1**, 291 (1919) *48*

H^+

OH^- **III-2112**

The heat of the reaction of hydrogen ions with hydroxyl ions was calculated at room temperature.

$$H^+ + OH^- \rightarrow H_2O$$

E. Brinkman, R. Margaria, and F. J. W. Roughton, Trans. Roy. Soc. (London), **232A**, 65 (1934) *105*

HCl

Ag **III-2113**

Hydrochloric acid was standardized volumetrically by metallic silver by the method of Stas.

$$(2HCl + 2Ag \rightarrow 2AgCl + H_2)$$

V. H. Veley and J. J. Manley, Trans. Roy Soc. (London), **191**, 372 (1898) *105*

HCl

CCl_3CHO **III-2114**

When hydrogen chloride gas reacts with chloral a crystalline addition product, trichloroethylidene-chlorohydrine is formed.

$$CCl_3 \cdot CHO + HCl \rightarrow CCl_3 \cdot CH(OH)Cl$$

E. Mulder, Rec. trav. Chim., **7**, 332 (1888) *89*

HCl

$CH_3CHOHCH_2COONa$ **III-2115**

When the sodium salt of β-hydroxybutyric acid is treated with hydrochloric acid, β-hydroxy-butyric acid is formed.

$$CH_3CHOHCH_2COONa + HCl \rightarrow CH_3CHOHCH_2COOH + (NaCl)$$

Wislicenus, Ann., **149**, 205 (1869)
Ref., P. Latham, Biochem. J., **3**, 216 (1908) *53*

HCl

$CH_3SCH_2CH_2CH(NH_2)CN$ **III-2116**

When α-amino-γ-methylthiol-butyronitrile is boiled with concentrated hydrochloric acid, α-amino-γ-methylthiol-butyric acid is formed.

$$CH_3SCH_2CH_2CH(NH_2)CN + HCl + 2H_2O \rightarrow CH_3SCH_2CH_2CHNH_2COOH + (NH_4Cl)$$

Barger and Coyne, Biochem. J., **22**, 1421 (1928) *53*

HCl

$KClO_3$ **III-2117**

Dry HCl gas attacks potassium chlorate as follows:

$$2KClO_3 + 4HCl \rightarrow 2ClO_2 + 2H_2O + (2KCl) + (Cl_2)$$

J. W. Thomas, J. Chem. Soc. (London), **33**, 372 (1878) *91*

HCl

$KMnO_4$ **III-2118**

Potassium permanganate reacts violently with dry HCl gas evolving heat.

(a) $2KMnO_4 + 8HCl \rightarrow 2KCl + 2MnO_2 + 4H_2O + 3Cl_2$

(b) $MnO_2 + 4HCl \rightarrow MnCl_2 + Cl_2 + 2H_2O$

J. W. Thomas, J. Chem. Soc. (London), **33**, 372 (1878) *91*

HCl

KNO_3 **III-2119**

Dry hydrogen chloride gas attacks a number of metallic nitrates according to the following type of reaction.
Nitrates of Na, Bi, Cd, Co, Cu, Pb, Li, Mg, Mn, Hg, Ni, Ag, U, and Zn are similarly attacked. Heat is evolved in all cases and NOCl is sometimes formed, if a large excess of HCl is used.

$$2KNO_3 + 8HCl \rightarrow 2KCl + 2NO + 4H_2O + 3Cl_2$$

J. W. Thomas, J. Chem. Soc. (London), **33**, 369–371 (1878) *91*

HCl

$(KSbOC_4H_4O_6)_2 \cdot H_2O$ **III-2120**

Potassium antimonyl tartrate is attacked by dry hydrogen chloride.

$$(KSbOC_4H_4O_6)_2 \cdot H_2O + 6HCl \rightarrow 2SbCl_3 + 2KHC_4H_4O_6 + 3H_2O$$

Thomas, J. Chem. Soc. (London), **33**, 372 (1878) *91*

HCl

K_2CrO_4 **III-2121**

Dry hydrogen chloride gas attacks potassium chromate evolving heat as follows:

(a) $2K_2CrO_4 + 2HCl \rightarrow K_2Cr_2O_7 + 2KCl + H_2O$

(b) $K_2Cr_2O_7 + 2HCl \rightarrow 2KCl + H_2CrO_4 + (CrO_3)$

(c) $2H_2CrO_4 + 12HCl \rightarrow 2CrCl_3 + 8H_2O + 3Cl_2$

J. W. Thomas, J. Chem. Soc. (London), **33**, 371 (1878) *91*

HCl

$K_3C_6H_5O_7 \cdot H_2O$ **III-2122**

Potassium citrate is attacked by dry HCl as follows:

(a) $K_3C_6H_5O_7 \cdot H_2O + HCl \rightarrow KCl + K_2HC_6H_5O_7 + H_2O$

(b) $K_2HC_6H_5O_7 + HCl \rightarrow KCl + KH_2C_6H_5O_7$

J. W. Thomas, J. Chem. Soc. (London), **33**, 371 (1878) *91*

HCl

$K_4Fe(CN)_6$ **III-2123**

Potassium ferrocyanide is attacked by dry HCl gas, heat being evolved.

$$K_4Fe(CN)_6 + 4HCl \rightarrow H_4Fe(CN)_6 + 4KCl$$

J. W. Thomas, J. Chem. Soc. (London), **33**, 372 (1878) *91*

HCl

III-2124

NH_3

Dry hydrogen chloride in benzene reacts with ammonia (dried by lime and by phosphorus pentoxide) to form an immediate precipitate of white ammonium chloride. Ionization is not involved. Benzene vapors likewise catalyze the union of hydrogen chloride and ammonia.

$$HCl + NH_3 \rightarrow \underline{NH_4Cl}$$

L. Kahlenberg, J. Phys. Chem., **6**, 1 (1902) *7*

HCl

III-2125

$(NH_4)_2MoO_4$

Ammonium molybdate is attacked by dry HCl gas. Much heat is evolved. A small amount of chlorine and MoO_2 are formed.

(a) $(NH_4)_2MoO_4 + 2HCl \rightarrow MoO_3 + 2NH_4Cl + H_2O$

(b) $MoO_3 + 2HCl \rightarrow MoO_2 + Cl_2 + H_2O$

J. W. Thomas, J. Chem. Soc. (London), **33**, 372 (1878) *91*

HCl

III-2126

$NaHSO_4$

Sodium hydrogen sulfate is decomposed by dry HCl gas.

$$NaHSO_4 + HCl \rightarrow NaCl + H_2O + SO_2 + (O)$$

J. W. Thomas, J. Chem. Soc. (London), **33**, 372 (1878) *91*

HCl

III-2127

NaOCl

Dry sodium hypochlorite is violently attacked by dry HCl, heat being evolved.

(a) $NaOCl + HCl \rightarrow HOCl + NaCl$

(b) $HOCl + HCl \rightarrow Cl_2 + H_2O$

J. W. Thomas, J. Chem. Soc. (London), **33**, 372 (1878) *91*

HCl

Na_2CO_3 **III-2128**

Hydrochloric acid was standardized volumetrically by sodium carbonate.

$$2HCl + Na_2CO_3 \rightarrow (2NaCl + H_2O + CO_2)$$

V. H. Veley and J. J. Manley, Trans. Roy. Soc. (London), **191**, 372 (1898) *105*

HCl

Na_2SO_4 **III-2129**

Dry HCl gas is absorbed by the dry sulfates of ammonium, sodium, and potassium.

$$Na_2SO_4 + HCl \rightarrow NaCl + NaHSO_4$$

J. W. Thomas, J. Chem. Soc (London), **33**, 372 (1878) *91*

HCl

$Na_2S_2O_3$ **III-2130**

Sodium thiosulfate is easily decomposed by dry HCl gas.

$$Na_2S_2O_3 + 2HCl \rightarrow S + SO_2 + H_2O + 2NaCl$$

J. W. Thomas, J. Chem. Soc. (London), **33**, 372 (1878) *91*

HCl

O_2 **III-2131**

The Deacon equilibrium has been calculated from spectroscopic data on chlorine.

$$4HCl + O_2 \rightarrow 2Cl_2 + 2H_2O$$

A. R. Gordon and C. Barnes, Trans. Roy. Soc. (Canada) [III], **26**, 171 (1932) *69*

HCl

$Pb_3(C_6H_5O_7)_2$ **III-2132**

Lead citrate is violently attacked by dry HCl. The organic portion of the molecule is attacked, empyreumatic vapors and charring being observed. Heat is liberated.

$$Pb_3(C_6H_5O_7)_2 + 6HCl \rightarrow 3PbCl_2 + H_2O + ?$$

J. W. Thomas, J. Chem. Soc. (London), **33**, 371 (1878) *91*

HCl

III-2133

Sn_4P_3

Hydrochloric acid attacks the tin in the reagent and forms a mixture of phosphines and hydrogen.

$$Sn_4P_3 + HCl \rightarrow PH_3 + P_2H_4 + H_2 + ?$$

P. Jolibois, Compt. rend., **148**, 637 (1909) *38*

HCl

III-2134

ε

Hydrochloric acid solution was electrolyzed to produce hydrogen and chlorine. The purpose was to study the combination of hydrogen and chlorine under the action of light.

$$2HCl \leftrightharpoons H_2 + Cl_2$$

P. V. Bevan, Trans. Roy. Soc. (London), **202**, 78 (1904) *105*

HCl

III-2135

ε

Hydrochloric acid was electrolyzed to make hydrogen and chlorine.

$$2HCl \rightarrow H_2 + Cl_2$$

H. B. Dixon, Trans. Roy. Soc. (London), **184**, 168 (1893) *105*

$HClO_4$

Br_2 **III-2136**

Bromine when shaken with an aqueous solution of perchloric acid, forms perbromic acid.

$$2HClO_4 + Br_2 \rightarrow 2HBrO_4 + Cl_2$$

M. M. P. Muir,
Ref., R. Gerstl, Ber., **7**, 259 (1874) *11*

HCN

CO

NH_3

III-2137

The catalytic formation of hydrogen cyanide is more complex than given by the equation:

$$CO + NH_3 \rightarrow HCN + H_2O$$

G. Bredig, E. Elöd, and G. Kortüm, Z. Elektrochem., **36**, 1007 (1930) *86*

HCN

O_2

III-2138

The catalytic oxidation of hydrogen cyanide with CuO, NiO, Fe_2O_3, Bi_2O_3 and Pt is studied.

$$4HCN + 7O_2 \rightarrow 2H_2O + 4NO + 4CO_2$$

B. Neumann and G. Manke, Z. Elektrochem., **35**, 763 (1929) *86*

HI

$CCl_3 \cdot CHO$

III-2139

When hydriodic acid reacts with chloral, a liquid product, trichloroethylideneiodohydrine, is formed.

$$CCl_3 \cdot CHO + HI \rightarrow CCl_3 \cdot CH(OH)I$$

M. E. Mulder, Rec. trav. Chim., **7**, 332 (1888) *89*

HI

$CCl_3 \cdot CHO$

III-2140

Chloral reacts with gaseous hydrogen iodide forming iodal and hydrogen chloride.

$$CCl_3 \cdot CHO + 3HI \rightarrow CI_3 \cdot CHO + 3HCl$$

M. E. Mulder, Rec. trav. Chim., **7**, 310 (1888)
Ref., M. Bertrand, Jahresber., 588 (1881) *89*

HI

$(CN)_2$ **III-2141**

Cyanogen gas when passed into boiling concentrated hydriodic acid forms amino acetic acid, ammonium iodide and iodine.

$$(CN)_2 + 5HI + 2H_2O \rightarrow CH_2NH_2COOH + NH_4I + 2I_2$$

A. Emmerling, Ber. **6**, 1352 (1873) *11*

HI

HNO_2 **III-2142**

The oxidation of hydriodic acid by nitrous acid gives nitric oxide:

$$2HI + 2HNO_2 \rightarrow I_2 + 2H_2O + 2NO$$

A. Cambron and G. S. Whitby, Can. J. Res., **2**, 144 (1930) *69*

HI

H_2O_2 **III-2143**

In studying rates of reaction, hydrogen peroxide was allowed to react with hydriodic acid, forming iodine.

$$H_2O_2 + 2HI \rightarrow \underline{I_2} + 2H_2O$$

A. Vernon Harcourt and William Esson, Trans. Roy. Soc. (London), **186**, 817 (1895) *105*

HI

I_2 **III-2144**

Various complexes are formed when hydrogen iodide and iodine are dissolved in certain solvents, (water, sulfur dioxide and organic solvents):

(a) $$HI + I_2 \rightarrow HI_3$$

(b) $$HI_3 + I_2 \rightarrow HI_5$$

A. Berthoud and M. Mosset, J. Chim. phys., **33**, 272 (1936) *69*

HI

O_2 **III-2145**

Light

When carried out in the dark, the oxidation of hydrogen iodide in solution by free oxygen proceeds at a different speed than in the light.

$$4HI + O_2 \rightarrow 2I_2 + 2H_2O$$

Winter, Z. physik. Chem., **113**, 275 (1926)
Ref., A. Berthoud and G. Nicolet, J. Chim. phys., **25**, 163 (1928) *69*

HI

N-dimethylaminoacetic acid **III-2146**

When an aqueous solution of N-dimethylaminoacetic acid and excess hydriodic acid is concentrated, n-dimethylaminoacetic acid monohydriodide is formed. If less hydriodic acid is used, a semihydriodide is formed.

(a) $CH_2N(CH_3)_2COOH + HI \rightarrow CH_2N(CH_3)_2COOH \cdot HI$

(b) $2CH_2N(CH_3)_2COOH + HI + H_2O \rightarrow (CH_2N(CH_3)_2COOH)_2 \cdot HI \cdot H_2O$

Anslow and King, Biochem. J., **22**, 1258 (1928) *53*

HIO_3

P **III-2147**

A concentrated solution of iodic acid reacts vigorously with yellow phosphorus, forming phosphoric acid and free iodine.

$$2P + 2HIO_3 + 2H_2O \rightarrow 2H_3PO_4 + I_2$$

Million,
Ref., Ch. Friedel, Ber., **3**, 325 (1870) *11*

HIO_3

P **III-2148**

A dilute solution of iodic acid reacts with yellow phosphorus to form phosphoric acid and hydrogen iodide.

$$6P + 5HIO_3 + 9H_2O \rightarrow 6H_3PO_4 + 5HI$$

Million,
Ref., Ch. Friedel, Ber., **3**, 325 (1870) *11*

$HNOSO_4$

NaCl **III-2149**

Nitrosyl sulfuric acid when warmed with sodium chloride forms nitrosyl chloride and sodium acid sulfate.

$$HNOSO_4 + NaCl \rightarrow NOCl + NaHSO_4$$

Tilden,
Ref., R. Gerstl, Ber., **7**, 598 (1874) *11*

HNO_3

P_2O_5 **III-2150**

Nitrogen pentoxide is prepared by adding 71 parts of phosphorus pentoxide to 63 parts of nitric acid, keeping the temperature below 0°C, until the phosphoric anhydride has been added, and then warming the mixture in a crucible.

$$P_2O_5 + 2HNO_3 \rightarrow 2HPO_3 + N_2O_5$$

Berthelot,
Ref., A. Henninger, Ber., **6**, 1560 (1873) *11*

HSCN

H_2O **III-2151**

In the presence of sulfuric acid water acts on thiocyanic acid yielding carbon oxysulfide and ammonia. The thiocyanic acid is best freshly made from ammonium thiocyanate and sulfuric acid.

(a) $(2NH_4SCN + H_2SO_4 \rightarrow (NH_4)_2SO_4 + 2HSCN)$

(b) $HSCN + H_2O \rightarrow COS + NH_3$

P. Claesson, K. Sven. Vet. Ak. Bih. **9**, No. 14, 6 (1884) *10*

HSO_3^-

H_2 **III-2152**

During electrolysis of cobalt and nickel tartrate solutions containing bisulfite, the deposition of sulfur at the cathode was observed.

$$(4H + 4HSO_3^- \rightarrow 2\underline{S} + 2H_2O + 4OH^- + 2SO_2 + O_2)$$

J. E. Root, J. Phys. Chem., **9**, 1 (1905) *7*

HSO_3NO_2

PCl_5 **III-2153**

Phosphorus penta-chloride reacts with nitrosyl sulfuric acid with the evolution of heat and the formation of chlorosulfonic acid, nitrosyl chloride and phosphorus oxychloride.

$$HSO_3NO_2 + PCl_5 \rightarrow HSO_3Cl + NOCl + POCl_3$$

A. Michaelis and O. Schumann, Ber., **7**, 1076 (1874) *11*

HSO_3NO_2

Δ **III-2154**

Nitrosyl sulfuric acid begins to decompose at 30°C. The first reaction is probably that of dehydration, in the second (b) the water reacts with nitrosyl sulfuric acid to form sulfuric acid and nitrogen tri-oxide.

(a) $$2HSO_3NO_2 \rightarrow S_2O_5(NO_2)_2 + H_2O$$

(b) $$2HSO_3NO_2 + H_2O \rightarrow 2H_2SO_4 + N_2O_3$$

A. Michaelis and O. Schumann, Ber., **7**, 1077 (1874) *11*

H_2

AgCl **III-2155**

Silver chloride heated in a stream of hydrogen will give metallic silver and hydrochloric acid.

$$2AgCl + H_2 \rightarrow 2HCl + 2Ag$$

Tommasi, Gazz. chim. ital., **8**, 195 (1878) *21*

H_2

Br_2 **III-2156**

When a mixture of hydrogen and bromine are ignited the elements combine to form hydrogen bromide.

$$H_2 + Br_2 \rightarrow 2HBr$$

V. Mertz and W. Weith, Ber., **6**, 1519 (1873) *11*

H_2

III-2157

C

Methane is produced with 73–96% yield by treating purified sugar charcoal at 1150° with a current of pure dried hydrogen.

$$C + 2H_2 \rightarrow CH_4$$

W. A. Bone and H. F. Coward, J. Chem. Soc. (London), **93**, 1975 (1908)
Ref., ibid **97**, 1219 (1910) *57*

H_2

III-2158

$CH_3C_6H_4OH$

The cresols at 500° and an initial hydrogen pressure of 100 atm. in the absence of a catalyst yield phenol, benzene and toluene.

$$3CH_3C_6H_4OH + 4H_2 \rightarrow C_6H_5OH + C_7H_8 + C_6H_6 + 2H_2O + 2CH_4$$

C. M. Cawley, Fuel, **12**, 35 (1933) *75*

H_2

III-2159

CO

H_2O

The water gas catalytic process for producing hydrogen consists of the oxidation at 500° C of the carbon monoxide in water gas by steam in the presence of a catalyst (oxide of iron).

$$H_2 + CO + H_2O \rightarrow 2H_2 + CO_2$$

Hugh S. Taylor, Chem. & Met. Eng., **27**, 1263 (1922) *44*

H_2

III-2160

CO_2

The formation of methane from carbon dioxide and hydrogen is accompanied by diminution of volume, hence it will be favored by high pressure. High temperature works the other way, from the free-energy data it appears that, at 1000°C and atmospheric pressure the equilibrium inclines to the side of carbon dioxide, even in the presence of a large excess of hydrogen. Below 300°C practically all the carbon dioxide should go into methane. At about 600° the four compounds should be in molar equilibrium.

$$CO_2 + 4H_2 \leftrightarrows CH_4 + 2H_2O$$

H. N. Russell, Science, **81**, 1 (1935) *44*

H_2

CO_2 **III-2161**

In the presence of certain bacteria, no free hydrogen results from the fermentation of glycerol by marine anaerobes because it may be activated to unite with carbon dioxide with the production of methane.

$$CO_2 + 4H_2 \rightarrow CH_4 + 2H_2O$$

Claude E. Zobell, Science, **102**, 364 (1945) *44*

H_2

CO_2 **III-2162**

CO

When carbon dioxide and carbon monoxide are reduced by hydrogen in the presence of reduced nickel, cobalt or copper, methane is formed.

$$CO_2 + CO + 7H_2 \rightarrow 2CH_4 + 3H_2O$$

Sabatier and Senderens, Bull. soc. chim. (France), **27**, 704–88 (1902)
Ref., G. Guillemin and B. Delachanal, Compt. rend., **147**, 1310 (1908) *38*

H_2

CS_2 **III-2163**

When a well dried mixture of hydrogen and carbon disulfide is passed over hot platinum sponge, carbon and hydrogen sulfide are formed.

$$2H_2 + CS_2 \rightarrow C + 2H_2S$$

Alph. Cossa, Ber., **1**, 117 (1868) *11*

H_2

$C_6H_4(CO)_2O$ **III-2164**

At 450° and an initial pressure of hydrogen of 100 atm. without catalyst, phthalic anhydride produces water, toluene, benzene, some high boiling residue, carbon dioxide and carbon monoxide.

$$2C_6H_4(CO)_2O + 4H_2 \rightarrow C_6H_6 + C_6H_5CH_3 + 2CO_2 + CO + H_2O$$

C. M. Cawley, Fuel, **12**, 368 (1933) *75*

H_2

III-2165

$C_6H_4(OH)_2$

Resorcinol, catechol and quinol when hydrogenated in the presence of charcoal impregnated with ammonium molybdate and sulfur at 450° and an initial hydrogen pressure of 100 atm., yield mainly water, benzene and saturated hydrocarbons containing cyclohexane.

(a) $$C_6H_4(OH)_2 + 2H_2 \rightarrow C_6H_6 + 2H_2O$$

(b) $$C_6H_6 + 3H_2 \rightarrow C_6H_{12}$$

C. M. Cawley, Fuel, **12**, 35 (1933) *75*

H_2

III-2166

$C_6H_5COOC_2H_5$

At 115–200° and 100 atm. pressure of hydrogen and in the presence of a nickel catalyst, ethyl benzoate is hydrogenated to ethyl hexahydrobenzoate.

$$C_6H_5COOC_2H_5 + 3H_2 \rightarrow C_6H_{11}COOC_2H_5$$

Komatsu and Mitsui, Mem. Coll. Sci., Kyoto Imp. Univ., (Japan) **14**, 297A (1931)
Ref., C. M. Cawley, Fuel, **12**, 366 (1933) *75*

H_2

III-2167

C_6H_5COOH

At 350–400° and an initial pressure of hydrogen of 100 atm. without catalyst, benzoic acid produces water, toluene and a high boiling neutral oil mainly dibenzyl.

$$3C_6H_5COOH + 8H_2 \rightarrow C_6H_5CH_3 + (C_6H_5CH_2)_2 + 6H_2O$$

C. M. Cawley, Fuel, **12**, 367 (1933) *75*

H_2

III-2168

C_6H_5COOH

At 450° in the presence of activated charcoal containing ammonium molybdate and an initial pressure of hydrogen of 100 atm., benzoic acid is converted into water, benzene and toluene together with a small amount of saturated and unsaturated hydrocarbons.

$$2C_6H_5COOH + 7H_2 \rightarrow C_6H_6 + C_6H_5CH_3 + 4H_2O + CH_4$$

C. M. Cawley, Fuel, **12**, 367 (1933) *75*

H_2

C_6H_5COONa **III-2169**

Sodium benzoate was hydrogenated at 450° and an initial pressure of hydrogen of 100 atm. In the absence of a catalyst, sodium carbonate, benzene, toluene, water and a high boiling neutral oil mainly diphenylmethane were obtained. By the use of ammonium molybdate catalyst supported on charcoal, the same products were obtained but the yields of benzene and toluene were increased while the yield of neutral oil was decreased.

$$2C_6H_5COONa + 3H_2 \rightarrow Na_2CO_3 + C_6H_6 + C_7H_8 + H_2O$$

C. M. Cawley, Fuel, **12**, 368 (1933) *75*

H_2

$C_6H_5NH_2$ **III-2170**

At 760° in a tinned iron tube, hydrogen decomposes aniline yielding benzene and ammonia.

$$C_6H_5NH_2 + H_2 \rightarrow C_6H_6 + NH_3$$

E. Audibert and A. Raineau. Rev. ind. minerale (1923)
Ref., Fuel, **4**, 362 (1925) *75*

H_2

C_6H_5OH **III-2171**

Phenol at 340° under an initial pressure of hydrogen of 100 atm. and in the presence of cobalt sulfide is converted to cyclohexane, cyclohexanol being formed intermediately.

(a) $$C_6H_5OH + 3H_2 \rightarrow C_6H_{11}OH$$

(b) $$C_6H_{11}OH + H_2 \rightarrow C_6H_{12} + H_2O$$

Roberti, Ann de chim. appl. **21**, 217 (1931) *75*

H_2

C_6H_5OH **III-2172**

When phenol is hydrogenated under an initial hydrogen pressure of 100 atm., after 14 hours heating at 245° in the presence of nickel oxide as catalyst, hexahydrophenol is produced.

$$C_6H_5OH + 3H_2 \rightarrow C_6H_{11}OH$$

Ipatieff, J. Russ. Chem. Soc., **39**, 693
Ref., C. M. Cawley, Fuel, **11**, 217 (1932) *75*

H_2

C_6H_5OH **III-2173**

At about 800°, a mixture of phenol and hydrogen in a tinned iron tube reached a state of equilibrium.

$$2C_6H_5OH + 2H_2 \leftrightarrows 2C_6H_6 + 2H_2O$$

E. Audibert and A. Raineau, Rev. ind. minerale (1923)
Ref., Fuel, **4**, 362 (1925) *75*

H_2

C_6H_5OH **III-2174**

Phenol is converted into hydrocarbons consisting mainly of benzene and cyclohexane by treatment with hydrogen at 450° and an initial pressure of hydrogen of 100 atm. in the presence of charcoal impregnated with ammonium molybdate.

(a) $$C_6H_5OH + H_2 \rightarrow C_6H_6 + H_2O$$

(b) $$C_6H_6 + 3H_2 \rightarrow C_6H_{12}$$

(c) $$C_6H_5OH + 3H_2 \rightarrow C_6H_{11}OH$$

(d) $$C_6H_{11}OH + H_2 \rightarrow C_6H_{12} + H_2O$$

C. M. Cawley, Fuel, **11**, 221 (1932) *75*

H_2

$(C_6H_5)_2$ **III-2175**

At 760° in a tinned iron tube, hydrogen decomposes diphenyl yielding benzene.

$$C_6H_5 \cdot C_6H_5 + H_2 \rightarrow 2C_6H_6$$

E. Audibert and A. Raineau, Rev. ind. minerale (1923)
Ref., Fuel, **4**, 363 (1925) *75*

H_2

$(C_6H_5)_2CH_2$ **III-2176**

At 760° in a tinned iron tube, diphenylmethane was decomposed by hydrogen yielding benzene and methane.

$$C_6H_5 \cdot CH_2 \cdot C_6H_5 + 2H_2 \rightarrow 2C_6H_6 + CH_4$$

E. Audibert and A. Raineau, Rev. ind. minerale (1923)
Ref., Fuel, **4**, 363 (1925) *75*

H_2

$(C_6H_5)_2CO$ **III-2177**

At 450° and an initial pressure of hydrogen of 100 atm. in the presence of active charcoal containing ammonium molybdate, benzophenone was converted to benzene, toluene, water and a high boiling neutral oil, chiefly diphenylmethane.

$$2C_6H_5COC_6H_5 + 5H_2 \rightarrow C_6H_6 + C_6H_5CH_3 + (C_6H_5)_2CH_2 + 2H_2O$$

C. M. Cawley, Fuel, **12**, 368 (1933) *75*

H_2

$(C_6H_5)_2NH$ **III-2178**

At 760° in a tinned iron tube, diphenylamine was decomposed by hydrogen to benzene and ammonia.

$$C_6H_5 \cdot NH \cdot C_6H_5 + 2H_2 \rightarrow 2C_6H_6 + NH_3$$

E. Audibert and A. Raineau, Rev. ind. minerale (1923)
Ref., Fuel, **4**, 363 (1925) *75*

H_2

C_9H_8 **III-2179**

At 800–860° in a tinned iron tube, indene and hydrogen reach an equilibrium with benzene and propane.

$$C_9H_8 + 3H_2 \leftrightarrows C_6H_6 + C_3H_8$$

E. Audibert and A. Raineau, Rev. ind.minerale (1923)
Ref., Fuel, **4**, 363 (1925) *75*

H_2

$C_{10}H_7OH$ **III-2180**

At 450° and an initial hydrogen pressure of 100 atm. in the presence of active charcoal containing ammonium molybdate, β naphthol yields principally naphthalene and tetralin. α Naphthol reacts in a similar manner.

(a) $$C_{10}H_7OH + H_2 \rightarrow C_{10}H_8 + H_2O$$

(b) $$C_{10}H_8 + 2H_2 \rightarrow C_{10}H_{12}$$

C. C. Hall, Fuel, **12**, 425 (1933) *75*

H_2

$C_{10}H_8$ **III-2181**

At 350–400°, naphthalene is hydrogenated to tetralin in the presence of activated charcoal containing ammonium molybdate employing 100 atm. initial pressure of hydrogen.

$$C_{10}H_8 + 2H_2 \rightarrow C_{10}H_{12}$$

C. C. Hall, Fuel, **12**, 90 (1933) *75*

H_2

$C_{10}H_{12}$ **III-2182**

At 500°, tetralin is converted to ethylbenzene, toluene, benzene and ethane in the presence of active charcoal containing ammonium molybdate employing 100 atm. initial hydrogen pressure. Butyl benzene is an intermediate product of the reaction.

(a) $$C_{10}H_{12} + H_2 \rightarrow C_6H_5 \cdot C_4H_9$$

(b) $$C_6H_5 \cdot C_4H_9 + H_2 \rightarrow C_6H_5 \cdot C_2H_5 + C_2H_6$$

(c) $$C_6H_5 \cdot C_2H_5 + H_2 \rightarrow C_6H_5CH_3 + CH_4$$

(d) $$C_6H_5CH_3 + H_2 \rightarrow C_6H_6 + CH_4$$

C. C. Hall, Fuel, **12**, 92 (1933) *75*

H_2

$C_{10}H_{12}$ **III-2183**

At 800–860° in a tinned iron tube, tetrahydronaphthalene and hydrogen reach an equilibrium with benzene and butane.

$$C_{10}H_{12} + 2H_2 \rightarrow C_6H_6 + C_4H_{10}$$

E. Audibert and A. Raineau, Rev. ind. minerale (1923)
Ref., Fuel, **4**, 363 (1925) *75*

H_2

Cl_2 **III-2184**

Equal volumes of chlorine and hydrogen were exploded.

$$H_2 + Cl_2 \rightarrow (2HCl)$$

H. B. Dixon, Trans. Roy. Soc. (London), **184**, 143 (1893) *105*

H_2

Cl_2 **III-2185**

Hydrogen and chlorine, prepared by the electrolysis of hydrogen chloride, were exposed to the action of light in actinometer.

$$H_2 + Cl_2 \rightarrow 2HCl$$

Pringsheim, Wied. Ann., **32**, 384 (1887)
G. Dyson and A. Harden, J. Chem. Soc. (London), **83**, 29 (1903)
Ref., Burgess and Chapman, J. Chem. Soc. (London), **89**, 1399 (1906) *109*

H_2

$CuCO_3$ **III-2186**

In reduction of a catalyst mixture of Cu- and Ni-carbonates, the catalyst, carried on kieselguhr, is treated with hydrogen for one to four hours at 180°–220°, at the usual pressure, using sunflower oil or cotton seed oil as the medium in which the catalyst is suspended. After decanting the oil, the catalyst is washed with fresh hot oil.

$$CuCO_3 + H_2 \rightarrow \underline{Cu} + CO_2 + H_2O$$

Masloboino Zhirovoe Delo, **13**, No. 4, 31 (1937)
Ref., Abstracts, Oil and Soap, **15**, 3, 77 (1938) *111*

H_2

III-2187

D_2

The isotopic equilibria in the hydrogen-hydrogen oxide system can be reduced to three equations which involve all the components.

(a) $$H_2 + D_2 \rightleftharpoons 2HD$$

(b) $$D_2O + H_2 \rightleftharpoons H_2O + D_2$$

(c) $$H_2 + HDO \rightleftharpoons HD + H_2O$$

R. H. Crist and G. A. Dalin, J. Chem. Phy., **2**, 735 (1934) *44*

H_2

III-2188

D_2

Whenever hydrogen is liberated from an aqueous solution containing heavy water a separation of the isotopes occurs, i.e., the isotopic composition of the gas is not the same as that of the liquid.

(a) $$H_2 + D_2 \rightarrow 2HD$$

(b) $$H_2O + D_2O \rightarrow 2HOD$$

(c) $$H_2O + HD \rightarrow HOD + H_2$$

A. Farkas and L. Farkas, J. Chem. Phy., **2**, 468 (1934) *44*

H_2

III-2189

$FeCl_3$

Nascent hydrogen reduces ferric chloride to ferrous chloride with the minimum of heat. Hydrochloric acid is also produced in the course of the reaction.

$$2FeCl_3 + H_2 \rightarrow 2FeCl_2 + 2HCl$$

Pizzi, Gazz. chim. ital., **7**, 112 (1877) *21*

H_2

III-2190

HCNS

When hydrogen acts on thiocyanic acid, hydrocyanic acid and hydrogen sulfide are formed.

$$HCNS + H_2 \rightarrow HCN + H_2S$$

Gazz. chim. ital., **12**, 186 (1882) *21*

H_2

HCNS **III-2191**

If an excess of hydrogen is used on thiocyanic acid the product will be methyl sulfide and ammonia.

$$HCNS + 2H_2 \rightarrow CH_3S + NH_3$$

Gazz. chim. ital., **12**, 186 (1882) *21*

H_2

H_2SO_4 **III-2192**

Certain bacteria may cause the hydrogen from the fermentation of glycerol by marine anaerobes to reduce sulfates to hydrogen sulfide.

$$H_2SO_4 + 4H_2 \rightarrow H_2S + 4H_2O$$

Claude E. Zobell, Science, **102**, 364 (1945) *44*

H_2

N_2 **III-2193**

At ordinary temperature and in the absence of a catalyst, ammonia cannot be synthesized from its elements even at pressures up to 900 atmospheres.

$$N_2 + 3H_2 \rightarrow 2NH_3$$

E. Briner and A. Wroczynski, J. Chim. phys., **9**, 105 (1911) *69*

H_2

N_2 **III-2194**

$C_{10}H_8$

ε

When hydrogen, nitrogen and naphthalene (gaseous) are subjected to silent electrical discharge, hydrogen cyanide is formed.

$$C_{10}H_8 + H_2 + 5N_2 \rightarrow 10HCN$$

Franz Fischer and Kurt Peters, Brenn. chem., **10**, 113 (1929) *11*

H_2

III-2195

N_2O

ε

A mixture of hydrogen and nitrous oxide at room temperature and atmospheric pressure was exploded by an electric spark or by a detonator.

$$2H_2 + 2N_2O \rightarrow (2H_2O) + (2N_2)$$

W. A. Bone and R. P. Fraser. Trans. Roy. Soc. (London), **230A**, 363 (1932) *105*

H_2

III-2196

N_2O

One volume of nitrous oxide was exploded with hydrogen, forming water and nitrogen.

$$N_2O + H_2 \rightarrow (N_2 + H_2O)$$

H. B. Dixon, Trans. Roy. Soc. (London), **184**, 108 (1893) *105*

H_2

III-2197

N_2O

Hydrogen and nitrous oxide interact at the surface of a gold wire at 704° and 880° to give nitrogen and water.

$$H_2 + N_2O \rightarrow N_2 + H_2O$$

W. K. Hutchison and Cyril N. Hinshelwood, J. Chem. Soc. (London), p. 1557 (1926) *65*

H_2

III-2198

Na

Solutions of 18% or less sodium hydride in molten caustic may be produced by bubbling hydrogen into a fused caustic bath (700°F) containing molten sodium, the reactants being taken in suitable proportions for the concentration desired.

$$2Na + H_2 \rightarrow 2NaH$$

J. Albin, Iron Age, **156**, 60 (1945) *6*

H_2

Na **III-2199**

Hydrogen (at 1 atm. pressure) and sodium react rapidly and completely at 250–300°C when the liquid sodium is made to spread over an inert support or preformed sodium hydride. Best surface-active or dispersing agents are sodium derivatives of the more acidic aromatic hydrocarbons, such as anthracene, phenanthrene and fluorene. Fatty acid soaps of alkali and alkaline-earth metals are satisfactory under certain conditions.

$$Na + \tfrac{1}{2}H_2 \rightarrow NaH + 13.8 \text{ Cal.}$$

V. L. Hansley, U.S. Pat. 2,372,670 and 2,372,671 (April 3, 1945)
Ref., V. L. Hansley and P. J. Carlisle, Chem. & Eng. News, **23**, 1332 (1945) *6*

H_2

$Na_2C_2O_4$ **III-2200**

Electrolytic hydrogen acts on sodium oxalate to reduce it to water and sodium glycollate and this product with water gives sodium hydroxide and sodium hydroxyacetate.

(a) $$Na_2C_2O_4 + 2H_2 \rightarrow H_2O + CH_2ONa \cdot COONa$$

(b) $$CH_2ONa \cdot COONa + H_2O \rightarrow NaOH + CH_2OH \cdot COONa$$

Gazz. chim. ital., **12**, 195 (1882) *21*

H_2

$NiCO_3$ **III-2201**

In reduction of a catalyst mixture of Ni- and Cu-carbonates, the catalyst, carried on kieselguhr, is treated with hydrogen for one to four hours at 180°–220°, at the usual pressure, using sunflower oil or cotton seed oil as the suspending medium. After decanting the oil, the catalyst is washed with fresh hot oil.

$$NiCO_3 + H_2 \rightarrow \underline{Ni} + CO_2 + H_2O$$

Masloboino Zhirovoe Delo, **13**, No. 4, 31 (1937)
Ref., Abstracts, Oil and Soap, **15**, 3, 77 (1938) *111*

H_2

O **III-2202**

Direct union of oxygen and hydrogen takes place around 500°. The reaction occurs mostly at the surface of the (porcelain) vessel (a). Since hydrogen may be adsorbed, a probable product of the union of hydrogen with oxygen may be hydrogen peroxide (b). This latter compound, then may be an intermediate product in the mechanism of water formation (c).

(a) $$H_2 + \tfrac{1}{2}O_2 \rightarrow H_2O$$

(b) $$H_2 + O_2 \rightarrow H_2O_2$$

(c) $$H_2O_2 \rightarrow H_2O + \tfrac{1}{2}O_2$$

William C. McCullagh Lewis, J. Chem. Soc. (London), **115**:1, 190 (1919) *48*

H_2

OH **III-2203**

In order to determine the specific heat of water vapor at high temperatures the following heat of formation was needed.

$$OH + \tfrac{1}{2}H_2 \rightarrow H_2O + 63{,}550 \text{ Cal.}$$

R. W. Fenning and A. C. Whiffin, Trans. Roy. Soc. (London), **238A**, 151 (1940) *105*

H_2

O_2 **III-220**

In order to determine the specific heat of water vapor at high temperatures the following heat of formation was needed.

$$H_2 + \tfrac{1}{2}O_2 \rightarrow H_2O \text{ (gas)} + 57{,}660 \text{ Cal.}$$

R. W. Fenning and A. C. Whiffin, Trans. Roy. Soc. (London), **238A**, 151 (1940) *105*

H_2

O_2 **III-2205**

The homogeneous oxidation of hydrogen in a closed vessel is a chain reaction and proceeds according to the following steps:

(a) $$H_2 + O_2 \rightarrow 2OH$$

(b) $$H_2 + OH \rightarrow H_2O + H$$

(c) $$O_2 + H_2 + H \rightarrow H_2O + OH$$

Bonhoeffer and Haber, Z. physik. Chem., **137**, 337 (1928)
Ref., M. Prettre, J. Chim. phys., **33**, 189 (1936) *69*

H_2

O_2 **III-2206**

A mixture of hydrogen and oxygen at room temperature and atmospheric pressure was exploded electrically or by a detonator.

$$2H_2 + O_2 \rightarrow (2H_2O)$$

W. B. Bone and Fraser, Trans. Roy. Soc. (London), **230A**, 363 (1932)
W. A. Bone et. al., ibid., **235A**, 29 (1936)
W. A. Bone et. al., ibid., **206A**, 14 (1906)
F. P. Burt and E. C. Edgar, ibid., **216A**, 406 (1916)
W. T. David, ibid., **211A**, 392 (1912)
H. B. Dixon, ibid., **200**, 328 (1903)
H. B. Dixon, ibid., **184**, 107 (1893)
R. W. Fenning and A. C. Whiffin, ibid., **238A**, 151 (1940)
W. N. Hartley, ibid., **185**, 161 (1894)
Lord Kelvin, M. MacKean, and A. Galt, ibid., **191**, 213 (1898)
B. Lambert and D. J. Borgars, ibid., **237A**, 562 (1939)
G. D. Lieving and J. Dewar, ibid., **179**, 27 (1888)
Alexander Scott, ibid, **184**, 550 (1893) *105*

H_2

O_2 **III-2207**

Bac

Hydrogen is oxidized by certain bacteria to water.

$$H_2 + \tfrac{1}{2}O_2 \rightarrow H_2O + 68.4 \text{ Cal}$$

F. Fischer, R. Lieske, and K. Winzer, Brenn.-Chem., **11**, 453 (1930) *11*

H_2

III-2208

O_2

P

Hydrogen was mixed with air and exploded at an initial absolute pressure of 76.8 lbs. per sq. in. and at 50°C. The addition of as much as 15% CO and 35% air had little effect on the maximum pressure or the time to develop it.

$$2H_2 + O_2 \rightarrow (2H_2O)$$

R. W. Fenning., Trans. Roy. Soc. (London), **225A**, 331 (1925) *105*

H_2

III-2209

O_3

Mixtures of hydrogen, ozone and oxygen at 78° and 100° react as follows:

(a) $$2O_3 \rightarrow 3O_2$$

(b) $$H_2 + O_3 \rightarrow H_2O + O_2$$

J. W. Belton, and R. O. Griffith, and A. M. McKlown, J. Chem. Soc. (London), p. 3154 (1926) *66*

H_2

III-2210

O_3

Small quantities of hydrogen sulfide found in air are oxidized by the action of ozone.

$$H_2S + O_3 \rightarrow H_2O + S + O_2$$

Olsen and Ulrich, J. Ind. Eng. Chem., **6**, 620 (1914) *22*

H_2

III-2211

S

When dry H_2 is led through boiling sulfur, hydrogen sulfide is formed.

$$H_2 + S \rightarrow H_2S$$

Alph Cossa, Ber., **1**, 117 (1868) *11*

H_2

ε **III-2212**

Hydrogen free of oxygen may be activated by the vacuum tube discharge to give triatomic hydrogen if the walls are poisoned with stearic acid.

$$3H_2 \rightarrow 2H_3$$

Binder, Filby and Grubb, Can. J. Res., **4**, 331 (1931) *94*

H_2

Δ **III-2213**

The production of atomic hydrogen in discharge tubes has been studied and calorimetric measurements have been made on partially dissociated hydrogen from discharge tubes. From these measurements and from spectroscopic data the heat of dissociation of hydrogen at 300°K is calculated to be 103,680 cal./g.-mole.

$$H_2 \rightarrow 2H$$

H. G. Poole, Proc. Roy. Soc. (London), **163A**, 404 (1937) *110*

H_2

Δ **III-2214**

In order to determine the specific heat of hydrogen at high temperatures the following heat of formation was needed.

$$2H \rightarrow H_2 + 103{,}220 \text{ Cal.}$$

R. W. Fenning and A. C. Whiffens., Trans. Roy. Soc. (London), **238A**, 151 (1940) *105*

H_2

Δ **III-2215**

O_2

Mixtures of helium and hydrogen burn only if the concentration of the latter exceeds 28% in volume.

$$(2H_2 + O_2 \rightarrow 2H_2O)$$

J. Satterly and E. F. Burton, Trans. Roy. Soc. (Canada) [III], **13**, 211 (1919) *69*

H_2

Decanoic acid **III-2216**

Hydrogenation of the lighter unsaturated acids is easily accomplished by treatment with hydrogen at ordinary temperatures, in the presence of active platinum black. This acid, as well as other low-melting acids, is converted into a higher-melting compound which can be more readily purified for identification in analytical work.

$$H_2C{:}CH(CH_2)_7COOH + H_2 \rightarrow CH_3CH_2(CH_2)_7COOH$$

J. B. Brown, Oil and Soap, **13**, 12, 305 (1936) *111*

H_2

Light **III-2217**

Cl_2

The photosynthesis of hydrogen chloride takes place only with radiation of shorter wave length than 5000 Å.

$$H_2 + Cl_2 \rightarrow 2HCl$$

Taylor, Phil. Mag., **49**, 116 (1925)
Ref., J. Cathala, J. Chim. phys., **25**, 182 (1928) *69*

H_2

Linolin **III-2218**

Hydrogenation of linolin can be controlled by the variation of the ratio of Ni to soluble material in the catalysts. One of the products which can be thus controlled is olein.

$$[CH_3(CH_2)_4HC{:}CHCH_2HC{:}CH(CH_2)_7COO]_3C_3H_5 + 3H_2$$
$$\rightarrow [CH_3(CH_2)_7HC{:}CH(CH_2)_7COO]_3C_3H_5$$

Leslie G. Jenness, Ph. D., in "Nickel Catalysts"
Ref., Oil and Soap, **11**, 7, 131 (1934) *111*

H_2

Linolin **III-2219**

Hydrogenation of linolin can be controlled by variation of the ratio of Ni to soluble material in the catalysts. One of the products which may be thus controlled is Iso-olein (Elaidin).

$$[CH_3(CH_2)_4HC{:}CHCH_2HC{:}CH(CH_2)_7COO]_3C_3H_5 + 3H_2 \rightarrow [CH_3(CH_2)_7CH{:}HC(CH_2)_7COO]_3C_3H_5$$

Leslie G. Jenness, in "Nickel Catalysts"
Ref., Oil and Soap, **11**, 7, 131 (1934) *111*

H_2

Linolin **III-2220**

Hydrogenation at higher temps. decreases the formation of Iso-oleic (Elaidic) acid. Results: Temp. 250–300°, time, 57–176 min., Ni 1–10%, pressure, 25 mm water. Yield of Elaidic, 1.93 to 12.22 per cent. Reaction inhibited by higher temperature.

$$[CH_3(CH_2)_4HC{:}CHCH_2HC{:}CH(CH_2)_7COO]_3C_3H_5 + 3H_2 \rightarrow [CH_3(CH_2)_7HC{:}CH(CH_2)_7COO]_3C_3H_5$$

Karl Kammermeyer, Oil and Soap, **12**, 2, 33 (1935)
Ref., Sei-Ichi Ueno, J. Soc. Chem. Ind. (Japan), **37**, supp. 457, (1934) *111*

H_2

Palmitic Acid **III-2221**

In 1931, Adkins and Folkers found that with the proper catalyst, it was possible to obtain the corresponding alcohols having the same number of carbon atoms as the acid hydrogenated. Conditions necessary for this hydrogenation not stated in this article.

$$CH_3(CH_2)_{14}COOH + 2H_2 \rightarrow CH_3(CH_2)_{14}CH_2OH + H_2O$$

Adkins and Folkers, J. Am. Chem. Soc., **53**, 1095 (1931)
Ref., W. C. Gangloff, Oil and Soap, **15**, 1, 15 (1938) *111*

H_2

Ricinoleic acid **III-2222**

Castor oil was hardened to m.p. 62.0°–66.5°, and iodine number 3. The fat acids became predominantly stearic, with some other acids. The double bond was reduced more easily than the —OH group.

(a) $CH_3(CH_2)_5CHOHCH_2CH{:}CH(CH_2)_7COOH + H_2 \rightarrow CH_3(CH_2)_5CHOH(CH_2)_{10}COOH$

(b) $CH_3(CH_2)_5CHOH(CH_2)_{10}COOH + H_2 \rightarrow CH_3(CH_2)_{16}COOH + H_2O$

A. Bömer and Fr. Brehm, Z. Unters. Lebensm., **72**, 1–34 (1936)
Ref., Oil and Soap, **13**, 10, 266 (1936) *111*

H_2

Stearic Acid **III-2223**

Preparation of materials of hydrocarbon or alcohol character from esters of high mol. wt. acid by hydrogenation at 60 to 300 atm., and temps. over 250°.
(Reaction of free stearic acid used in order to simplify this abstract.)

(a) $C_{17}H_{35}COOH + H_2 \rightarrow C_{17}H_{35}CH_2OH + H_2O$

(b) $C_{17}H_{35}CH_2OH + H_2 \rightarrow C_{17}H_{35}CH_3 + H_2O$

Walter Schrauth, Ger. Pat. No. 629,244.
Ref., Oil and Soap, **13**, 11, 294 (1936) *111*

H_2O

Br_2 **III-2224**

Hypobromous acid is formed by action of bromine on water:

$$Br_2 + H_2O \rightarrow HBrO + HBr$$

A. Berthoud and M. Mosset, J. Chim. phys., **33**, 272 (1936) *69*

H_2O

$CH_3SCH_2CH_2CH(OC_2H_5)_2$ **III-2225**

HCl

β-Methylthiolpropaldehydediethylacetal is hydrolyzed by boiling for 30 minutes with very dilute hydrochloric acid, forming β-methylthiolpropaldehyde, which separates on cooling.

$$CH_3SCH_2CH_2CH(OC_2H_5)_2 + H_2O \rightarrow \underline{CH_3SCH_2CH_2CHO} + (2C_2H_5OH)$$

Barger and Coyne, Biochem. J., **22**, 1420 (1928) *68*

H_2O

$CH_3SCH_2CH(OC_2H_5)_2$ **III-2226**

HCl

When methylthiolacetal is boiled in 1% hydrochloric acid solution for 30 minutes, methylthiolacetaldehyde is formed.

$$CH_3SCH_2CH(OC_2H_5)_2 + H_2O \rightarrow CH_3SCH_2CHO + (2C_2H_5OH)$$

Barger and Coyne, Biochem. J., **22**, 1424 (1928) *68*

H_2O

$(CH_3CO)_2O$ **III-2227**

In preparation of truly anhydrous acetic acid, two liters of glacial acetic are refluxed with one hundred milliliters of acetic anhydride for three hours. Anhydrous acid thus prepared is to be stored in paraffin-sealed bottles.

$$(CH_3CO)_2O + H_2O \rightarrow 2CH_3COOH$$

W. S. Martin and R. C. Stillman, Oil and Soap, **10**, 30 (1933) *111*

H_2O

Cl **III-2228**

Chlorine in the nascent state reacts with water to give oxygen and hypochlorous acid.

(a) $$H_2O + 4Cl \rightarrow 2HCl + O_2$$

(b) $$H_2O + 2Cl \rightarrow HCl + HOCl$$

E. Doumer, J. Chim. phys., **19**, 38 (1921) *69*

H_2O **III-2229**

Cl

SO_2

Chlorine and sulfur dioxide react with water to form sulfuric acid and hydrochloric acid.

$$2Cl + SO_2 + 2H_2O \rightarrow H_2SO_4 + 2HCl$$

Greenwalt, J. Ind. Eng. Chem., **2**, 555 (1910) *22*

H_2O **III-2230**

Cl_2

Water vapor heated with chlorine to 120° forms oxygen and hydrochloric acid.

$$2H_2O + 2Cl_2 \rightarrow 4HCl + O_2$$

C. Baskerville and R. Stevenson, J. Ind. Eng. Chem., **3**, 471 (1911) *25*

H_2O **III-2231**

Fe

When iron comes in contact with oxygen-bearing solution it reacts with the oxygen and water, forming ferric hydroxide.

$$6H_2O + 3O_2 + 4Fe \rightarrow 4Fe(OH)_3$$

R. J. McKay, Ind. Eng. Chem., **17**, 24 (1925) *23*

H_2O **III-2232**

Fe

FeO

The high ratio of water gas consumed, to hydrogen produced, in the water gas catalytic process can be attributed to equilibrium reactions in both steaming and reducing phases.

(a) $3Fe + 3H_2O \rightleftharpoons 3FeO + 3H_2$

(b) $3FeO + H_2O \rightleftharpoons Fe_3O_4 + H_2$

(c) $3Fe + 3CO_2 \rightleftharpoons 3FeO + 3CO$

(d) $3FeO + CO_2 \rightleftharpoons Fe_3O_4 + CO$

Hugh S. Taylor, Chem. & Met. Eng., **27**, 1263 (1922) *44*

H_2O

Mg **III-2233**

Water vapor is reduced to hydrogen by magnesium.

(a) "HO + Mg → MgO + H"

(b) $H_2O + Mg \rightarrow MgO + H_2$

H. Cretier, Z. anal. Chem., **13**, 1 (1874) *28*

H_2O

Mg_3N_2 **III-2234**

The moisture content of a gas may be determined by the use of magnesium nitride and a determination of the ammonia liberated.

$$Mg_3N_2 + 6H_2O \rightarrow 3Mg(OH)_2 + 2NH_3$$

Harry Levin, Karl Uhrig, and F. M. Roberts, Anal. Ed., **17**, 212 (1945) *4*

H_2O

Na-Hg **III-2235**

The decomposition of water by sodium, magnesium or ammonium amalgams is accompanied by the emission of photons (2510Å):

$$2H_2O + 2Na\text{-}Hg \rightarrow 2NaOH + H_2 + \underline{2Hg}$$

R. Audubert, J. Chim. phys., **33**, 507 (1936) *69*

H_2O

O_2 **III-2236**

Hydrogen peroxide, 100% pure, liquid and free of water is made through electrolytical oxidation of water by means of silent electric discharge.

$$2H_2O + O_2 \rightarrow 2H_2O_2 + 44{,}320 \text{ cal}$$

P. M. Wolf, Z. Elektrochem., **20**, 204 (1914) *86*

H_2O **III-2237**

($RaBr_2$)

Radium bromide decomposes water into its component elements. Solid radium bromide left in contact with a mixture of oxygen and hydrogen slowly yields water.

$$(2H_2O \rightarrow 2H_2 + O_2)$$

Berger, Davis, and Edwards, J. Soc. Chem. Ind. (London), **24**, 266 (1905) *109*
Ref., W. Ramsay, J. Chem. Soc. (London), **91**, 931 (1907)

H_2O **III-2238**

Rn

Radium emanation (radon) in contact with water is converted to neon, while the water is decomposed.

$$2H_2O + Rn \rightarrow 2H_2 + O_2 + Ne$$

A. T. Cameron and Sir W. Ramsay, J. Chem. Soc. (London), **93**, 996 (1908) *57*

H_2O **III-2239**

S

When water vapor is passed through boiling sulfur, hydrogen sulfide and dithionic acid are formed.

$$3H_2O + 4S \rightarrow 2H_2S + H_2S_2O_3$$

Jacob Myres, Ber., **5**, 260 (1872) *11*

H_2O **III-2240**

S

When pure water in which is suspended finely divided sulfur is electrolized, a small amount of hydrogen sulfide is formed.

$$2S + 2H_2O \rightarrow 2H_2S + O_2$$

Alph. Cossa, Ber., **1**, 118 (1868) *11*

H_2O

Aminomalonicnitrile **III-2241**

When aminomalonicnitrile is warmed with an aqueous solution of barium hydroxide, glycocol, ammonia, and carbon dioxide are formed.

$$CH(NH_2)(CN)_2 + 4H_2O \rightarrow CH_2(NH_2)COOH + CO_2 + 2NH_3$$

P. Latham, Biochem. J., **3**, 199 (1908) *53*

H_2O

Association **III-2242**

Precise vapor density measurements were made on ethyl alcohol, methyl alcohol, water, and benzene. From these could be calculated the concentration of normal and associated water molecules.

$6H_2O \leftrightarrows$	$3(H_2O)_2 \leftrightarrows$	$2(H_2O)_3$	Wt. percent at
75	17	8	0.75°C.
74	26	0	30°C.

Earl of Berkeley and E. Stenhouse, Trans. Roy. Soc. (London), **229A**, 255 (1930) *105*

H_2O

β and γ Rays **III-2243**

Radium chloride in a sealed glass bulb is placed in a glass tube and plunged in distilled water contained in a vessel under vacuum. The glass filters out the alpha rays emitted by the radium salt; the beta and gamma rays react with water to form hydrogen gas and hydrogen peroxide.

$$2H_2O + \text{beta, gamma rays} \rightarrow H_2 + H_2O_2$$

M. Kernbaum, Compt. rend., **148**, 706 (1909) *38*

H_2O

Ca-Stearate **III-2244**

Calcium soaps formed in hard waters hydrolyze readily, allowing some free fatty acids to be present in the water, along with the weak hydroxides of the alkaline earth metals. This action impedes the action of the remaining alkali soap. Stearic salt used as example:

$$(C_{17}H_{35}COO)_2Ca + H_2O \rightarrow Ca(OH)_2 + 2C_{17}H_{35}COOH$$

W. C. Gangloff, Oil and Soap, **15**, 1, 15 (1938) *111*

H_2O

III-2245

Glycerol Stearate

"Water is passed in uninterrupted circulation through a column of molten fat at a temp. high enough and pressure high enough to liberate the fatty acid and glycerol. The glycerol is washed by the water and is evacuated as glycerol-containing water, the fat is evacuated at the other end."

Glycerol stearate reaction shown as example.

$$C_3H_5(C_{17}H_{35}COO)_3 + 3H_2O \rightarrow C_3H_5(OH)_3 + 3C_{17}H_{35}COOH$$

Henkel and Cie., G. m, b. H., F. Pat., Jan. 28, 1937, No. 808,069.
Ref., Abstracts of Patents, Oil and Soap, **14**, 11, 304 (1937) *111*

H_2O

III-2246

Mg-Stearate

Magnesium soaps formed in hard waters hydrolize readily, allowing some free fatty acids to be present in the water, along with the weak hydroxides of the alkaline earth metals. This action impedes the action of the remaining alkali soap. Stearic salt used as example.

$$(C_{17}H_{35}COO)_2Mg + 2H_2O \rightarrow Mg(OH)_2 + 2C_{17}H_{35}COOH$$

W. C. Gangloff, Oil and Soap, **15**, 1, 15 (1938) *111*

H_2O

III-2247

ε

A mixture made of approximately 88.6% of oxygen and 11.4% of hydrogen by weight results when an electric arc is formed between platinum electrodes under water.

$$2H_2O \rightarrow 2H_2 + O_2$$

F. Petřik, Chem. Ob., **9**, 43 (1934) *5*

H_2O

III-2248

ε

Water was electrolyzed to get pure hydrogen; barium hydroxide was used as an electrolyte.

$$2H_2O \rightarrow 2H_2 + O_2$$

H. B. Dixon and E. C. Edgar, Trans. Roy. Soc. (London), **205A**, 170 (1906)
W. A. Bone and R. V. Wheeler, ibid., **206A**, 12 (1906)
E. C. Edgar, ibid., **209A**, 1 (1909) *105*

H_2O_2

III-2249

Ag_2O

The oxidation silver oxide by hydrogen peroxide yields metallic silver, oxygen and water.

(a) $Ag_2O + 3H_2O_2 \rightarrow H_4Ag_2O_6 + H_2O$

(b) $H_4Ag_2O_6 \rightarrow 2Ag + 2H_2O + 2O_2$

J. H. Kastle and A. S. Loevenhart, Am. Chem. J., **29**, 575 (1903) *21*

H_2O_2

III-2250

(Au)

The decomposition of hydrogen peroxide on the surface of gold is a zero order reaction:

$$2H_2O_2 \rightarrow 2H_2O + O_2$$

P. A. Giguère and O. Maass, Can. J. Res., **B18**, 84 (1940) *69*

H_2O_2

III-2251

Br_2

In acid solution at room temperature hydrogen peroxide reduces bromine to bromide.

$$H_2O_2 + Br_2 \rightarrow O_2 + 2HBr$$

W. C. Bray and R. S. Livingston, J. Am. Chem. Soc., **45**, 1251 (1923) *16*

H_2O_2

III-2252

$(C_2H_5)_2SO_4$

An alkaline solution of hydrogen peroxide was treated with diethyl sulfate over a period of 20 hours at 0° to give ethyl hydrogen peroxide and diethyl peroxide in low yields. The procedure is modified from Baeyer and Villiger, Ber., **34**, 738 (1901).

(a) $H_2O_2 + (C_2H_5)_2SO_4 + KOH \rightarrow C_2H_5OOH + C_2H_5KSO_4 + H_2O$

(b) $H_2O_2 + 2(C_2H_5)_2SO_4 + 2KOH \rightarrow (C_2H_5)_2O_2 + 2C_2H_5KSO_4 + 2H_2O$

E. J. Harris, Proc. Roy. Soc. (London), **168A**, 1 (1938) *110*

H_2O_2

III-2253

n-$(C_3H_7)_2SO_4$

An alkaline solution of hydrogen peroxide was treated with di-n-propyl sulfate at 0° and stirred for 8 hours to give low yields of n-propyl hydrogen peroxide (b. 35°/2 cm.) and di-n-propyl peroxide (b. 51–53°/8 cm.). The procedure is similar to that used by Baeyer and Villiger, Ber., **34**, 738 (1901) with diethyl sulfate.

(a) $H_2O_2 + \text{n-}(C_3H_7)_2SO_4 + KOH \rightarrow \text{n-}C_3H_7OOH + \text{n-}C_3H_7KSO_4 + H_2O$

(b) $H_2O_2 + 2\text{n}-(C_3H_7)_2SO_4 + 2KOH \rightarrow \text{n-}(C_3H_7)_2O_2 + 2\text{n-}C_3H_7KSO_4 + 2H_2O$

E. J. Harris, Proc. Roy. Soc. (London), **173A**, 126 (1939) *110*

H_2O_2

III-2254

$C_7H_8N_2O$

A water solution of benzamidoxime to which has been added nickel chloride yields with oxygen or hydrogen peroxide in the presence of ammonia, violet-red needles.

$$3C_6H_5CN_2H_3O + NH_3 + 3H_2O_2 \rightarrow (C_6H_5C\begin{matrix}NH_2\\NO\end{matrix})_3N + 6H_2O$$

J. V. Dubský and M. Kuraš, Chem. List., **24**, 464 (1930) *5*

H_2O_2

III-2255

$Ce(SO_4)_2$

A recent method for determining hydrogen peroxide in the presence of organic matter is titration with ceric sulfate solutions.

$$2Ce(SO_4)_2 + H_2O_2 \rightarrow Ce_2(SO_4)_3 + H_2SO_4 + O_2$$

J. S. Reichert, S. A. McNeight, and H. W. Rudel, Anal. Ed., **11**, 194 (1939) *44*

H_2O_2

D_2O **III-2256**

When heavy water is added to hydrogen peroxide the following exchange reactions occur:

(a) $H_2O_2 + D_2O \rightarrow H_2O + D_2O_2$

(b) $H_2O_2 + D_2O_2 \rightarrow 2HDO_2$

(c) $H_2O + D_2O \rightarrow 2HDO$

Giguère and Maass, Can J., Res., **18B**, 89 (1940) *94*

H_2O_2

($Fe_2O_3 \cdot 2FeCl_3$) **III-2257**

The catalytic decomposition of hydrogen peroxide by ferric oxychloride is a reaction of the first order.

$$2H_2O_2 \rightarrow 2H_2O + O_2$$

J. Duclaux, J. Chim. phys., **20**, 18 (1923) *69*

H_2O_2

HBr **III-2258**

In acid solution at room temperature, hydrogen peroxide oxidizes bromide to bromine.

$$H_2O_2 + 2HBr \rightarrow Br_2 + 2H_2O$$

W. C. Bray and R. S. Livingston, J. Am. Chem. Soc., **45**, 1251 (1923) *16*

H_2O_2

HCOH **III-2259**

In the oxidation of formic aldehyde, the hydrogen peroxide adds to the aldehyde in its highest proportion forming the holoxide condition of oxidation and then breaks down into its highest stable oxidation product.

$$HCOH + H_2O_2 \rightarrow HCOOH + H_2O$$

J. H. Kastle and A. S. Loevenhart, Am. Chem. J., **29**, 569 (1903) *21*

H_2O_2

III-2260

HCl

Chlorine is evolved when dry hydrogen chloride is passed into anhydrous hydrogen peroxide even at low temperatures.

$$2HCl + H_2O_2 \rightarrow Cl_2 + (2H_2O)$$

O. Maass and P. G. Hiebert, J. Am. Chem. Soc., **46**, 290 (1924) *1*

H_2O_2

III-2261

HNO_2

On treating nitrous acid with 3% hydrogen peroxide, a pernitric acid would be formed (a) which would slowly decompose after (b):

(a) $$HNO_2 + 2H_2O_2 \rightarrow HNO_4 + 2H_2O \ (?)$$

(b) $$HNO_4 + H_2O \rightarrow HNO_3 + H_2O_2$$

F. Raschig, Z. angew. Chem., **17**, 1419 (1904)
Ref., W. H. Hatcher and D. W. MacLauchlan, Can. J. Res., **B16**, 253 (1938) *69*

H_2O_2

III-2262

H_2CO_3

Hydrogen peroxide oxidizes carbonic acid to percarbonic acid.

$$H_2O_2 + H_2CO_3 \rightarrow H_2CO_4 + H_2O$$

M. LeBlanc and R. Zellmann, Z. Elektrochem., **29**, 182 (1923) *86*

H_2O_2

III-2263

H_2SO_3

In the oxidation of sulfurous acid the hydrogen peroxide adds right on to the acid in exactly the proportion of its highest labile, holoxide condition of oxidation and then breaks down into the highest stable oxidation product.

(a) $$H_2SO_3 + H_2O_2 \rightarrow H_4SO_5$$

(b) $$H_4SO_5 \rightarrow H_2SO_4 + H_2O$$

J. H. Kastle and A. S. Loevenhart, Am. Chem. J., **29**, 569 (1903) *21*

H_2O_2

Hg **III-2264**

The mechanism for the decomposition of H_2O_2 on a mercury surface is undoubtedly as follows:

(a) $2Hg + H_2O_2 \rightarrow Hg_2O + H_2O$

(b) $Hg_2O + 2H_2O_2 \rightarrow 2HgO + 2H_2O + O$

(c) $O + H_2O_2 \rightarrow H_2O + O_2$

L. Elder and E. K. Rideal, Trans. Faraday Soc., **23**, 551 (1927) *85*

H_2O_2

KI **III-2265**

When hydrogen peroxide in acid solution is added to an iodide, iodine is liberated quantitatively.

$$2KI + H_2SO_4 + H_2O_2 \rightarrow K_2SO_4 + 2H_2O + I_2$$

C. T. Kingzett, Analyst, **13**, 62 (1888) *40*

H_2O_2

$KMnO_4$ **III-2266**

Hydrogen peroxide reacts with potassium permanganate in acid solution with the evolution of oxygen.

$$2KMnO_4 + 5H_2O_2 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 8H_2O + 5O_2$$

Carpenter and Nicholson, Analyst, **9**, 37 (1884) *40*

H_2O_2

$KMnO_4$
H_2SO_4 **III-2267**

Hydrogen peroxide reacts with potassium permanganate with the aid of sulfuric acid to form potassium sulfate, manganese sulfate water and oxygen.

$$5H_2O_2 + 2KMnO_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 8H_2O + 5O_2$$

Bach, Am. Chem. J., **29**, 465 (1903) *44*
J. S. Reichert, S. A. McNeight and H. W. Rudel, Anal. Ed., **11**, 194 (1939) *21*
Aschoff, J. f. prakt. Chem., **81**, 404 (1850) *1*
Ref., J. Am. Chem. Soc., **1**, 321 (1876)

H_2O_2 **III-2268**

C_6H_7N

$K_2Cr_2O_7$

Bach's test for hydrogen peroxide is made as follows: Add 2–3 drops of 5% oxalic acid to equal parts of the solution to be tested and a 0.003% solution of potassium dichromate containing 6 drops of anilin per litre. The presence of hydrogen peroxide, one part in 200,000, is detected by a pink color.

(a) $(2K_2Cr_2O_7 + 10H_2O_2 \rightarrow 4H_3CrO_7 + 4KOH + 2H_2O)$?

(b) $(H_3CrO_7 + C_6H_7N \rightarrow C_6H_7N \cdot H_3CrO_7)$?

Bach, Compt. rend., **119**, 1218 (1894) *69*
Ref., G. W. Shearer, Trans. Roy. Soc. (Canada) [III] **2**, 171 (1908)

H_2O_2 **III-2269**

$K_2S_2O_8$

Hydrogen peroxide reacts slowly with potassium persulfate under ordinary conditions.

$$H_2O_2 + K_2S_2O_8 \rightarrow K_2SO_4 + H_2SO_4 + O_2$$

Friend, Proc. Chem. Soc. (London), **20**, 65, 198, J. Chem. Soc. (London), **85**, 597, 1533 *1*
Ref., J. Am. Chem. Soc., **27**, 1345 (1907)

H_2O_2 **III-2270**

NH_4CNS

In the oxidation of ammonium thiocyanate, the hydrogen peroxide adds to the thiocyanate in the proportion of its highest labile state forming an unstable compound which breaks down to its highest oxidation product.

(a) $NH_4CNS + 3H_2O_2 \rightarrow NH_4CNS \cdot 3H_2O_2$

(b) $NH_4CNS \cdot 3H_2O_2 \rightarrow NH_4SO_4 + HCN + 2H_2O$

A. S. Kastle and J. H. Loevenhart, Am. Chem. J., **29**, 569 (1903) *21*

H_2O_2

N_2O_5 **III-2271**

At low temperature pure hydrogen peroxide and nitrogen pentoxide behave as a peracid:

$$H_2O_2 + N_2O_5 \rightarrow HNO_3 + HNO_4$$

J. N. Friend, Text-book of Inorganic Chemistry, III (1931)
Ref., W. H. Hatcher and D. W. MacLauchlan, Can. J. Res., **B16**, 253 (1938) *69*

H_2O_2

NaOBr **III-2272**

Hydrogen peroxide decomposes sodium hypobromite with the formation of oxygen and sodium bromide.

$$NaOBr + H_2O_2 \rightarrow NaBr + H_2O + O_2$$

W. M. Dehn, J. Am. Chem. Soc., **29**, 1316 (1907)
Ref., ibid., **31**, 526 (1909) *1*

H_2O_2

$Pb(C_2H_3O_2)OH$ **III-2273**

Basic lead acetate is oxidized to lead dioxide by very dilutesolutions of hydrogen peroxide.

$$H_2O_2 + Pb(C_2H_3O_2)OH \rightarrow PbO_2 + CH_3COOH + H_2O$$

C. F. Schoenbein, Z. anal. Chem., **1**, 440 (1862) *28*

H_2O_2

(Pt) **III-2274**

Platinum black readily decomposes H_2O_2 yielding oxygen and water.

$$2H_2O_2 + (Pt) \rightarrow 2H_2O + O_2$$

Mulder and Van Der Meulen, Rec. trav. Chim., **1**, 171 (1882) *39*

H_2O_2 **III-2275**

$SnCl_2$

HCl

Stannous chloride reacts quantitatively with hydrogen peroxide.

$$SnCl_2 + 2HCl + H_2O_2 \rightarrow SnCl_4 + 2H_2O$$

J. S. Reichert, S. A. McNeight, and H. W. Rudel, Anal. Ed., **11**, 194 (1939) *44*

H_2O_2 **III-2276**

$TiCl_3$

HCl

One of the oldest methods for the determination of hydrogen peroxide in the presence of organic matter is based on the oxidation of titanium trichloride solution by hydrogen peroxide.

$$2TiCl_3 + H_2O_2 + 2HCl \rightarrow 2TiCl_4 + 2H_2O$$

J. S. Reichert, S. A. McNeight, and H. W. Rudel, Anal. Ed., **11**, 194 (1939) *44*

H_2O_2 **III-2277**

Blood catalase

At 40° blood catalase catalyzes the decomposition of hydrogen peroxide.

$$2H_2O_2 \rightarrow 2H_2O + O_2$$

K. Nosoka, J. Biochem. (Japan), **8**, 301 (1941) *61*

H_2O_2 **III-2278**

Catalyst-Pt

In the catalytic decomposition of hydrogen peroxide by colloidal platinum, only one molecule of the peroxide enters into reaction.

$$H_2O_2 \rightarrow H_2O + O$$

Bredig and von Berneck, J. Phys. Chem., **31**, 258 (1899)
Ref., J. H. Loevenhart and A. S. Kastle, Am. Chem. J., **29**, 399 (1903)
Ref., Traube, ibid., **29**, 437 (1903) *21*

H_2O_2

Cystine **III-2279**

Hydrogen peroxide reacts with cystine under physiological conditions to form taurine.

$$(5H_2O_2 + [SCH_2CH(NH_2)COOH]_2 \rightarrow 2CO_2 + 4H_2O + 2H_2NCH_2CH_2SO_3H)$$

A. Schöberl, Z. physiol. Chem., **216**, 193 (1933) *14*

H_2O_2

Glutaminyl monoanilide **III-2280**

Glutaminyl monoanilide is oxidized to succinyl monoanilide when warmed with H_2O_2 and a trace of $FeCl_3$ in ammoniacal solution as a catalyst.

$$NH_4OOCCHNH_2(CH_2)_2CONHC_6H_5 + H_2O_2 \rightarrow (2H_2O) + NH_4OOCCH_2CH_2COC_6H_5NH + HCN$$

Stewart and Tunnicliffe, Biochem. J., **19**, 207 (1925) *53*

H_2O_2

n-heptoic acid **III-2281**

When heptoic acid and hydrogen peroxide are heated in ammoniacal solution for four hours, some methyl-n-butyl ketone, acetic acid, γ-keto-n-heptoic acid, and δ-keto-n-heptoic acid are formed.

$$(3C_6H_{13}COONH_4 + 3H_2O_2 \rightarrow CH_3COC_4H_9 + CH_3COONH_4 + C_3H_7COCH_2CH_2COONH_4 + C_2H_5CO(CH_2)_3COONH_4 + 3H_2O)$$

P. W. Clutterbuck and H. S. Raper, Biochem. J., **19**, 385 (1925) *53*

H_2O_2

III-2282

n-hexoic acid

When n-hexoic acid and hydrogen peroxide are heated in ammoniacal solution for 4 hours, some n-propyl-methyl ketone, acetic acid, γ-keto-n-hexoic acid and δ-keto-n-hexoic acid are formed.

$$4C_5H_{11}COONH_4 + H_2O_2 \rightarrow 2CH_3COC_3H_7 + CH_3COONH_4 + C_2H_5COCH_2CH_2COONH_4 + CH_3CO(CH_2)_3COONH_4(+ H_2O + ?)$$

P. W. Clutterbuck and H. S. Raper, Biochem. J., **19**, 385 (1925) *53*

H_2O_2

III-2283

Light

The photochemical decomposition of hydrogen peroxide is inhibited by dilute solutions of sulfuric acid, sodium chloride, acetanilide, calcium hydroxide, sodium hydroxide, barium hydroxide, phosphoric acid, acetic acid, benzoic acid, and tartaric acid:

$$2H_2O_2 \rightarrow 2H_2O + O_2$$

J. H. Mathews and H. A. Curtis, J. Phys. Chem., **18**, 166 and 521 (1914) *7*

H_2O_2

III-2284

Metals

Hydrogen peroxide decomposes with metals to give the following equations:

(a) $$H_2O_2 + M \rightarrow M^+ + OH + OH^-$$

(b) $$H_2O_2 + OH \rightarrow H_2O + HO_2$$

(c) $$O_2 + H_2O_2 \rightarrow OH^- + OH + O_2$$

Weiss, Trans. Faraday Soc., **31**, 1547 (1935)
Giguère and Maass, Can. J. Res., **18B**, 89 (1940) *94*

H_2O_2

Myristic Acid **III-2285**

When myristic acid and hydrogen peroxide are heated in ammoniacal solution for 4 hours, some γ-keto-myristic acid is formed.

$$C_{13}H_{27}COONH_4 + 3H_2O_2 \rightarrow C_{10}H_{21}COCH_2CH_2COONH_4 + (5H_2O)$$

P. W. Clutterbuck and H. S. Raper, Biochem. J., **19**, 385 (1925) *53*

H_2O_2

n-octoic acid **III-2286**

When n-octoic acid and H_2O_2 are heated in ammoniacal solution for 4 hours, some methyl-n-amyl ketone, acetic acid, γ-keto-n-octoic acid, and δ-keto-n-octoic acid are produced.

$$(3C_7H_{15}COONH_4 + H_2O_2 \rightarrow CH_3COC_5H_{11} + CH_3COONH_4 + C_4H_9COCH_2CH_2COONH_4 + C_3H_7CO(CH_2)_3COONH_4 + 2H_2O)$$

P. W. Clutterbuck and H. S. Raper, Biochem. J., **19**, 385 (1925) *53*

H_2O_2

Oleic acid **III-2287**

In preparation of dihydroxystearic acid, oleic acid is treated in glacial acetic acid or acetone (at about 60°C), with hydrogen peroxide.

$$CH_3(CH_2)_7HC{:}CH(CH_2)_7COOH + 2HO - OH \rightarrow CH_3(CH_2)_7HCOH - HCOH(CH_2)_7COOH + 2HOH$$

Hilditch, J. Chem. Soc. (London), p. 1828 (1926)
Ref., Ralston and Bauer, Oil and Soap, **13**, 7, 171 (1936) *111*

H_2O_2

Palmitic acid **III-2288**

When palmitic acid and H_2O_2 are heated in ammonical solution for four hours, some γ-keto-palmitic acid is produced.

$$C_{15}H_{31}COONH_4 + 2H_2O_2 \rightarrow C_{12}H_{25}COCH_2CH_2COONH_4 + (3H_2O)$$

P. W. Clutterbuck and H. S. Raper, Biochem. J., **19**, 385 (1925) *53*

H_2O_2

III-2289

Stearic acid

When stearic acid and H_2O_2 are heated in ammoniacal solution for 4 hours, methyl-quindecyl ketone and γ-keto-stearic acid (1%) are obtained.

$$C_{17}H_{35}COONH_4 + H_2O_2 \rightarrow CH_3COC_{15}H_{31} + (H_2O) + C_{14}H_{29}COCH_2CH_2COONH_4 + ?$$

P. W. Clutterbuck and H. S. Raper, Biochem. J., **19**, 385 (1925) *53*

H_2O_2

III-2290

Uric acid

If hydrogen peroxide reacts with uric acid under physiological conditions, allantoin is formed besides some cyanuric acid and some carbonyl diurea.

$$H_2O_2 + C_5H_4N_4O_3 \rightarrow CO_2 + C_4H_6N_4O_3$$

H. Wieland and T. F. Macrae, Z. physiol. Chem., **203**, 83 (1931) *14*

H_2O_2

III-2291

Decomp.

Two weakly held hydrogen atoms of one molecule of the hydrogen peroxide split the second molecule into two hydroxyl groups producing water and oxygen.

$$2H_2O_2 \rightarrow 2H_2O + O_2$$

Traube, Ber., **26**, 1476 (1893)
Ref., J. H. Loevenhart and A. S. Kastle, Am. Chem. J., **29**, 425 (1903) *21*

H_2O_2

III-2292

Decomp.

Hydrogen peroxide reverts to the oxide as it liberates its active oxygen content.

$$2H_2O_2 \rightarrow 2H_2O + O_2$$

J. S. Reichert, S. A. McNeight, and H. W. Rudel, Anal. Ed., **11**, 194 (1939) *44*

H_2O_2

Decomp. **III-2293**

The decomposition of alkaline hydrogen peroxide on Pyrex glass is a first order reaction:

$$2H_2O_2 \rightarrow 2H_2O + O_2$$

P. A. Giguère and O. Maass, Can. J. Res., **B18**, 84 (1940) *69*

H_2S

C_3H_6 **III-2294**

At temperatures around 200°C and in presence of various catalysts, propylene reacts with hydrogen sulfide to form propyl mercaptan. The reaction does not go to completion; the percent of yield depending on the type of catalyst.

$$(H_2S + C_3H_6 \rightarrow CH_3CH_2 \cdot CH_2 \cdot SH)$$

H. R. Duffy, R. D. Snow, and D. B. Keyes, Ind. Eng. Chem., **26**, 93 (1934) *24*

H_2S

$FeCl_3$ **III-2295**

O_2

Hydrogen sulfide passed into ferric chloride solution precipitates sulfur and leaves ferrous chloride and hydrochloric acid in solution. This solution on exposure to air or oxygen reverts the ferrous chloride to ferric chloride.

$$2FeCl_3 + H_2S \rightarrow 2FeCl_2 + 2HCl + \underline{S}$$

K. E. Peterson, Sv. Kem. Tidsk., **1**, 125 (1889) *10*

H_2S

$Fe(OH)_3$ **III-2296**

O_2

Indirect oxidation of hydrogen sulfide and ferric hydroxide in air yields ferric sulfide and water in the first stage and ferric hydroxide and free sulfur in the second stage.

(a) $$2Fe(OH)_3 + 3H_2S \rightarrow Fe_2S_3 + 6H_2O$$

(b) $$2Fe_2S_3 + 6H_2O + 3O_2 \rightarrow 4Fe(OH)_3 + 6S$$

K. E. Peterson, Sv. Kem. Tidsk., **1**, 125 (1889) *10*

H_2S **III-2297**

H_2O

Hydrogen sulfide forms, with water, a complex that undergoes electrolytic oxidation:

$$H_2S + nH_2O \rightarrow H_2S \cdot nH_2O \rightarrow H^+ + HS \cdot nH_2O^-$$

R. H. Wright and O. Maass, Can. J. Res., **6**, 588 (1932) *69*

H_2S **III-2298**

I_2

A simultaneous determination of hydrogen sulfide and carbon dioxide in a continuous gas stream consists of titrating the former with standard iodine and the latter with standard barium hydroxide solution, using starch and phenolphthalein as the respective indicators.

$$H_2S + I_2 \rightarrow 2HI + S$$

Clyde L. Blohm and Fred C. Riesenfeld, Anal. Ed., **18**, 373 (1946) *44*

H_2S **III-2299**

I_2

H_2O

When hydrogen sulfide is passed into an aqueous solution and suspension of iodine, hydrogen iodide and sulfur are formed. (The reaction does not take place in the absence of water.)

$$I_2 + H_2S \rightarrow 2HI + S$$

Alex. Naumann, Ber., **2**, 180 (1869) *11*

H_2S **III-2300**

O

NH_3

The process of manufacturing ammonium salts from illuminating gas is passing hydrogen sulfide over bog iron heated to 60–70°C. The sulfur dioxide gas reacts with ammonia and water forming ammonium acid sulfite.

(a) $$H_2S + 3O \rightarrow H_2O + SO_2$$

(b) $$SO_2 + NH_3 + H_2O \rightarrow NH_4HSO_3$$

K. Burkheiser, J. Ind. Eng. Chem., **2**, 557 (1910) *22*

H_2S

O_2 **III-2301**

A mixture of hydrogen sulfide and oxygen at room temperature and atmospheric pressure was exploded by an electric spark or by a detonator.

$$2H_2S + 3O_2 \rightarrow (2H_2O) + (2SO_2)$$

W. A. Bone and R. P. Fraser, Trans. Roy. Soc. (London), **230A**, 363–85 (1932) *105*

H_2S

O_2 **III-2302**

Hydrogen sulfide is oxidized to free sulfur at temperatures above 300°C with approximately 75% conversion.

$$2H_2S + O_2 \rightarrow 2\underline{S} + 2H_2O$$

W. H. Cadman, Ind. Eng. Chem., **26**, 316 (1934) *24*

H_2S

O_2 **III-2303**

Bac.

Hydrogen sulfide is oxidized by certain bacteria, first to sulfur and then to sulfuric acid.

(a) $$H_2S + \tfrac{1}{2}O_2 \rightarrow H_2O + S$$

(b) $$S + 1\tfrac{1}{2}O_2 + H_2O \rightarrow H_2SO_4 + 141 \text{ Cal.}$$

F. Fischer, R. Lieske, K. Winzer, Brenn. Chem., **11**, 453 (1930) *11*

H_2SO_4

Δ **III-2304**

Sulfuric acid produced oxygen by passing it over porcelain in a hot tube.

$$H_2SO_4 \rightarrow H_2S + 2O_2$$

C. Baskerville and R. Stevenson, J. Ind. Eng. Chem., **3**, 471 (1911) *22*

$H_2S_2O_3$

III-2305

$AgNO_3$

Thiosulfuric acid reacts with a solution of silver nitrate to form silver sulfide, sulfuric and nitric acids.

$$H_2S_2O_3 + 2AgNO_3 + H_2O \rightarrow Ag_2S + H_2SO_4 + 2HNO_3$$

Jacob Myers, Ber., **5**, 261 (1871) *11*

$H_2S_4O_6$

III-2306

$Hg(CN)_2$

H_2O

Mercuric cyanide when added to tetrathionic acid forms sulfuric acid, mercuric sulfide, free sulfur, and hydrocyanic acid.

$$H_2S_4O_6 + Hg(CN)_2 + 2H_2O \rightarrow 2H_2SO_4 + HgS + S + 2HCN$$

D. Mendelejeff, V. von Richter, Ber., **3**, 872 (1870) *11*

H_3PO_2

III-2307

O_2

Hypophosphorous acid is oxidized when allowed to stand a long time in contact with the air, to phosphorus acid.

$$(H_3PO_2 + O \rightarrow H_3PO_3)$$

C. Rammelsberg, Ber., **1**, 185 (1868) *11*

KOH

III-2308

MnO_2

O_2

When manganese dioxide and potassium hydroxide are heated to 450°C potassium manganate and water are formed.

$$2KOH + MnO_2 + \tfrac{1}{2}O_2 \rightarrow K_2MnO_4 + H_2O$$

H. Vogel, Ber., **3**, 902 (1870) *11*

NH_3

$Hg(NO_3)_2$ **III-2309**

A yellow compound resembling basic nitrate separates out when mercuric nitrate is added to liquid ammonia.

$$Hg(NO_3)_2 + 2NH_3 \rightarrow \underline{(HgNH_2NO_3)} + (NH_4NO_3)$$

H. P. Cady, J. Phys. Chem., **1**, 711 (1896–7) 7

NH_3

ε **III-2310**

(NaCl)

The formation of ammonium and amine when a solution of liquid ammonia containing a dissolved sodium compound is electrolyzed, as suggested by Bleekrode, Phil Mag. (5), **5**, 384 (1878), is doubted and experimental evidence is adduced against it.

$$2NH_3 \rightarrow NH_4 + NH_2$$

H. P. Cady, J. Phys. Chem., **1**, 708 (1896–7) 7

N_2H_4

$Mn_2(SO_4)_3$ **III-2311**

An incomplete mono-de-electronation of hydrazine by manganic ion may be represented by the following equation.

$$2N_2H_4 + Mn_2(SO_4)_3 \rightarrow N_2 + 2NH_3 + 2MnSO_4 + H_2SO_4$$

A. G. Houpt, K. W. Sherk, and A. W. Browne, Anal. Ed., **7**, 54 (1935) 44

N_2H_4

O **III-2312**

The permanganate oxidation of hydrazine in hydrochloric acid solution corresponds quantitatively to the equation.

$$N_2H_4 + 2O \rightarrow N_2 + 2H_2O$$

A. G. Houpt, K. W. Sherk, and A. W. Browne, Anal. Ed., **7**, 54 (1935) 44

N_2H_4

III-2313

O

The presence of ammonia in the permanganate oxidation products of hydrazine points toward the conclusion that about one-third of the nitrogen of the hydrazine is converted to ammonia in accordance with the tentative equation.

$$3N_2H_4 + 3O \rightarrow 2N_2 + 2NH_3 + 3H_2O$$

A. G. Houpt, K. W. Sherk, and A. W. Browne, Anal. Ed., **7**, 54 (1935) *44*

$NaHSO_3$

III-2314

Zn

A concentrated sodium bisulfite solution when treated with zinc in the absence of air forms sodium hydrogen hyposulfite.

$$NaHSO_3 + Zn \rightarrow NaHSO_2 + (ZnO)$$

Schutzenberger,
Ref., Ch. Friedel, Ber., **2**, 431 (1869) *11*

Il_2O_3

HNO_3 **III-2315**

$Pb(NO_3)_2$

The double lead nitrate seems to offer an efficient method for the concentration of Illinium.

$$(Il_2O_3 + 6HNO_3 + 3Pb(NO_3)_2 \rightarrow 3Pb(NO_3)_2 \cdot 2Il(NO_3)_3 + 3H_2O)$$

D. W. Pearce and J. A. Harris, Trans. Roy. Soc. (Canada), [III], **24**, 145 (1930) *69*

$(CH_3)_3In$

III-2316

O_2

Oxidation of trimethyl indium takes place at low temperature.

$$4(CH_3)_3In + O_2 \rightarrow 2[(CH_3)_2In]_2O + 2C_2H_6$$

H. Gilman and R. G. Jones, J. Am. Chem. Soc., **62**, 2353 (1940) *16*

$(CH_3)_3In$

III-2317

O_2

When indium trimethyl is allowed to stand in contact with oxygen at $-78°$ for 250 hours the gaseous mixture was found to contain 8% ethane.

$$4In(CH_3)_3 + O_2 \rightarrow 2[In(CH_3)_2]_2O + 2C_2H_6$$

L. M. Dennis, R. W. Work and E. G. Rochow, J. Am. Chem. Soc., **56**, 1049 (1934) *1*

$(C_6H_5)_3In$

III-2318

O_2

Passing oxygen for 48 hours through a rapidly stirred benzene solution of triphenylindium oxidizes this compound and yields diphenyl.

$$2(C_6H_5)_3In + \tfrac{1}{2}O_2 \rightarrow [(C_6H_5)_2In]_2O + C_6H_5 \cdot C_6H_5$$

H. Gilman and R. G. Jones, J. Am. Chem. Soc., **62**, 2356 (1940) *16*

In

III-2319

Br

By passing bromine over heated indium for a short period, red colored indium monobromide is formed. If bromine reacts with indium for a longer period, dark yellow colored indium dibromide is obtained.

(a) $$In + Br \rightarrow \underline{InBr}$$

(b) $$In + 2Br \rightarrow \underline{InBr_2}$$

A. Thiel, Z. anorg. Chem., **40**, 280 (1904) *28*

In

$(C_6H_5)_2Hg$ **III-2320**

In an atmosphere of pure, dry nitrogen, the heating of diphenylmercury and indium cut into small pieces at a temperature of 138° for at least 48 hours produced triphenylindium.

$$2In + 3(C_6H_5)_2Hg \rightarrow 2(C_6H_5)_3\,In + 3Hg$$

H. Gilman and R. G. Jones, J. Am. Chem. Soc., **62**, 2353, 2355 (1940) *16*

In

Cl_2 **III-2321**

Indium chloride is produced when indium mixed with sugar carbon is heated in a current of chlorine.

$$In + Cl_2 \rightarrow InCl_2$$

Shin'ichirô Hakomori, J. Am. Chem. Soc., **52**, 2373 (1930) *1*

In

Cl_2 **III-2322**

Anhydrous indium trichloride is prepared by passing a stream of dry chlorine diluted with dry carbon dioxide over pieces of 99.98% pure indium metal heated just below its melting point. An apparently stepwise reaction yields lustrous plates of the trichloride.

$$(3Cl_2 + 2In \rightarrow 2InCl_3)$$

T. Moeller, J. Am. Chem. Soc., **63**, 1206 (1941) *3*

In

HCl **III-2323**

Hydrochloric acid acts on indium with the formation of indium trichloride.

$$2In + 6HCl \rightarrow 2InCl_3 + (3H_2)$$

L. F. Nilson and O. Pettersson, K. Sven. Vet. Ak. Handl. Bih. **14**, II, No. 6, 5 (1888) *10*

In

III-2324

HCl

Dry hydrogen chloride when passed over hot indium in the absence of air and moisture gives indium dichloride.

$$In + 2HCl \rightarrow InCl_2 + H_2$$

L. F. Nilson and O. Pettersson, K. Sven. Vet. Ak. Handl. Bih., **14**, II, No. 6, 8 (1888) *10*

In

III-2325

HCl

H_2SO_4

Indium metal dissolves in dilute hydrochloric acid or sulfuric acid with evolution of hydrogen and the formation of trivalent indium ions.

$$2In + 6H^+ \rightarrow 2In^{+++} + 3H_2$$

Treadwell and Hall, Analytical Chemistry, 5th Ed., **1**, 538 *93*

In

III-2326

$HClO_4$

Indium perchlorate separates in small crystals when indium metal is dissolved in dilute perchloric acid.

$$2In + 6HClO_4 + 16HOH \rightarrow 2In(ClO_4)_3 \cdot 8H_2O + (3H_2)$$

F. C. Mathers and C. G. Schluederberg, J. Am. Chem. Soc., **30**, 212 (1908) *1*

In

III-2327

HNO_3

NH_4OH

H_2SO_4

Indium sulfate is formed when the product produced by dissolving indium in nitric acid is precipitated with ammonium hydroxide and sulfuric acid added.

(a) $$2In + 6HNO_3 \rightarrow (2In(NO_3)_3 + 3H_2)$$

(b) $$In(NO_3)_3 + 3NH_4OH \rightarrow \underline{In(OH)_3} + (3NH_4NO_3)$$

(c) $$2In(OH)_3 + 3H_2SO_4 \rightarrow In_2(SO_4)_3 + (6H_2O)$$

L. M. Dennis, R. W. Work and E. G. Rochow, J. Am. Chem. Soc., **56**, 1047 (1934) *1*

In

$HgCl_2$ **III-2328**

$Hg(CH_3)_2$

Mercury dimethyl and mercuric chloride produce indium trimethyl when heated with indium at 100° for 8 days.

$$2In + 2HgCl_2 + 3Hg(CH_3)_2 \rightarrow 2In(CH_3)_3 + (4HgCl + Hg)$$

L. M. Dennis, R. W. Work and E. G. Rochow, J. Am. Chem. Soc., **56**, 1047 (1934) *1*

In

O_2 **III-2329**

Indium oxidizes slowly in contact with air.

$$4In + 3O_2 \rightarrow 2In_2O_3$$

Treadwell and Hall, Analytical Chemistry, 5th. Ed., **1**, p. 538 *93*

In^{+++}

KOH **III-2330**

Indium hydroxide is precipitated from solutions of In salts by KOH (or NH_4OH). The presence of tartaric acid prevents the precipitation.

$$In^{+++} + 3KOH \rightarrow \underline{In(OH)_3} + 3K^+$$

T. Richter, Compt. rend., **64**, 828 (1867) *29*

InBr

H_2O **III-2331**

Hot water will decompose indium monobromide into indium tribromide and indium.

$$3InBr + (H_2O) \rightarrow InBr_3 + 2In$$

A. Thiel, Z. anorg. Chem., **40**, 280 (1904) *28*

$InBr_2$

Δ **III-2332**

At a temperature of 1330°, indium dibromide is dissociated to 65% into indium monobromide and bromine.

$$2InBr_2 \rightleftarrows 2InBr + Br_2$$

A. Thiel, Z. anorg. Chem., **40**, 280 (1904) *28*

InCl

III-2333

H_2O

In the presence of water indium monochloride decomposes, yielding indium trichloride and metallic indium.

$$3InCl \rightarrow InCl_3 + 2In$$

L. F. Nilson and O. Pettersson, K. Sven. Vet. Ak. Handl. Bih., **14**, II, No. 6, 11 (1888) *10*

$InCl_2$

III-2334

H_2O

In the presence of water indium dichloride decomposes, yielding indium trichloride and metallic indium.

$$3InCl_2 + H_2O \rightarrow 2InCl_3 + In + H_2O$$

L. F. Nilson and O. Pettersson, K. Sven. Vet. Ak. Handl. Bih., **14**, II, No. 6, 8 (1888) *10*
Ref., F. C. Mathers, J. Am. Chem. Soc., **29**, 492 (1907) *1*

$InCl_2$

III-2335

In

When a mixture of indium dichloride and metallic indium is heated indium monochloride is formed.

$$InCl_2 + In \rightarrow 2InCl$$

L. F. Nilson and O. Pettersson, K. Sven. Vet. Ak. Handl. Bih., **14**, II, No. 6, 10 (1888) *10*

$InCl_3$

III-2336

$BaCO_3$

Barium carbonate causes the precipitation of a basic indium carbonate, even in the cold.

$$2InCl_3 + 2BaCO_3 + 2H_2O \rightarrow 2In(OH)CO_3 + (2BaCl_2 + 2HCl)$$

Treadwell and Hall, Analytical Chemistry, 5th. Ed., **1**, 539 *93*

$InCl_3$

H_2S **III-2337**

H_2S in neutral or acetic acid solutions gives a yellow precipitate of In_2S_3, which will not form, except on dilution, in the presence of mineral acids.

$$2In^{+++} + 3H_2S \rightarrow \underline{In_2S_3} + 6H^+$$

Treadwell and Hall, Analytical Chemistry, 5th ed., **1**, 539 *93*

$InCl_3$

KIO_3 **III-2338**

Indium iodate precipitates when solutions containing equivalent quantities of indium chloride and potassium iodate are mixed.

$$InCl_3 + 3KIO_3 \rightarrow \underline{In(IO_3)_3} + 3KCl.$$

F. C. Mathers and C. G. Schluederberg, J. Am. Chem. Soc., **30**, 212 (1908) *1*

$InCl_3$

KOH **III-2339**

In the absence of tartaric acid, the alkali hydroxides precipitate white, gelatinous indium hydroxide resembling aluminum hydroxide in appearance.

$$InCl_3 + 3KOH \rightarrow \underline{In(OH)_3} + (3KCl)$$

Treadwell and Hall, Analytical Chemistry, 5th. Ed., **1**, 538 *93*

$InCl_3$

K_2CrO_4 **III-2340**

Potassium chromate produces a yellow precipitate with indium salts, while $K_2Cr_2O_7$ does not form any.

$$3K_2CrO_4 + 2InCl_3 \rightarrow \underline{In_2(CrO_4)_3} + (6KCl)$$

Treadwell and Hall, Analytical Chemistry, 5th ed., **1**, 539 *93*

$InCl_3$

III-2341

$K_4Fe(CN)_6$

Indium salts give a white precipitate with $K_4Fe(CN)_6$, and none with $K_3Fe(CN)_6$.

$$4InCl_3 + 3K_4Fe(CN)_6 \rightarrow \underline{In_4[Fe(CN)_6]_3} + (12KCl)$$

Treadwell and Hall, Analytical Chemistry, 5th ed., **1**, 539 *93*

$InCl_3$

III-2342

MgO

H_2O

X-ray diffraction and analytical studies show that addition of magnesium oxide to an indium salt solution produces a precipitate of hydrous indium hydroxide rather than magnesium indate as claimed by Renz (Ber., **34**, 2763 (1901); **36**, 4394 (1903)).

$$2InCl_3 + 3MgO + 3H_2O \rightarrow \underline{2In(OH)_3} + 3MgCl_2$$

T. Moeller and J. G. Schnizlein, J. Phys. Chem., **51**, 771 (1947) *7*

$InCl_3$

III-2343

$(NH_4)_2CO_3$

Indium carbonate is precipitated by the addition of alkali carbonates to a solution of an indium salt. It dissolves in cold excess ammonium carbonate, but reprecipitates on boiling.

$$2InCl_3 + 3(NH_4)_2CO_3 \rightarrow \underline{In_2(CO_3)_3} + (6NH_4Cl)$$

Treadwell and Hall, Analytical Chemistry, 5th. Ed., **1**, 539 *93*

$InCl_3$

III-2344

NaOH

The precipitated oxide of indium consists of the hydrous trihydrate.

$$2InCl_3 + 6NaOH + aq. \rightarrow \underline{In_2O_3 \cdot 3H_2O \cdot aq.} + 6NaCl$$

A. B. Weiser and W. O. Milligan, J. Phys. Chem., **42**, 677 (1938) *85*

$InCl_3$

NaOH **III-2345**

Indium hydroxide is precipitated by the addition of sodium hydroxide in excess.

$$InCl_3 + 3NaOH \rightarrow \underline{In(OH)_3} + (3NaCl)$$

Hoffman, B. S. J. Res., **13**, 671 (1934) *93*

$InCl_3$

$Na_2C_2O_4$ **III-2346**

Sodium oxalate gives a white, crystalline precipitate of indium oxalate with indium salts.

$$2InCl_3 + 3Na_2C_2O_4 \rightarrow \underline{In_2(C_2O_4)_3} + (6NaCl)$$

Treadwell and Hall, Analytical Chemistry, 5th ed., **1**, 539 *93*

$InCl_3$

Na_2HPO_4 **III-2347**

Indium salts with Na_2HPO_4 give a white voluminous precipitate.

$$2InCl_3 + 3Na_2HPO_4 \rightarrow \underline{In_2(HPO_4)_3} + (6NaCl)$$

Treadwell and Hall, Analytical Chemistry, 5th ed., **1**, 539 *93*

$InCl_3$

$Na_2S_2O_4$ **III-2348**

Neutral aqueous solution of indium salts (chloride) are not precipitated by sodium hyposulfites in the cold but boiling causes the incomplete precipitation of indium sulfide. This is probably due to the decomposition of indium hyposulfite. Acidified solutions of indium salts do not precipitate with sodium hyposulfites.

(a) $$2InCl_3 + 3Na_2S_2O_4 \rightarrow In_2(S_2O_4)_3 + 6NaCl$$

(b) $$In_2(S_2O_4)_3 + 3Na_2S_2O_4 \rightarrow In_2S_3 + 3Na_2SO_4 + 6SO_2$$

O. Brunck, Ann., **336**, 281 (1904) *20*

$InCl_3$

III-2349

Na_2SeO_3

Sodium selenite precipitates from indium chloride solution amorphous, slimy 3:2:22 hydrated basic indium selenite.

$$8InCl_3 + 9Na_2SeO_3 + 28H_2O \rightarrow (18NaCl + 6HCl + \underline{(In_2(SeO_3)_3)_3 \cdot (In(OH)_3)_2 \cdot 22(H_2O)}$$

L. F. Nilson, Nova Acta. Soc. Uppsal. [3] **9**:7, 100 (1874) *10*

$InCl_3$

III-2350

Zn

Zinc metal precipitates indium from indium salts in the form of white, lustrous flakes.

$$2InCl_3 + 3Zn \rightarrow \underline{2In} + 3ZnCl_2$$

Treadwell and Hall, Analytical Chemistry, 5th ed., **1**, 539 *93*

$In(ClO_4)_3 \cdot 8H_2O$

III-2351

Δ

HNO_3

Indium oxide is formed when indium perchlorate is heated until decomposed and the resulting product converted into the nitrate by evaporating with nitric acid, and the resulting nitrate ignited.

(a) $In(ClO_4)_3 \cdot 8H_2O \rightarrow (InCl_3 + 6O_2 + 8H_2O)$

(b) $InCl_3 + 3HNO_3 \rightarrow In(NO_3)_3 + (3HCl)$

(c) $2In(NO_3)_3 + \Delta \rightarrow In_2O_3 + 3N_2O_5$

F. C. Mathers and C. G. Schluederberg, J. Am. Chem. Soc., **30**, 212 (1908) *1*

InF_3

III-2352

H_2

By heating indium fluoride gently in a stream of hydrogen, indium is formed.

$$2InF_3 + 3H_2 \rightarrow \underline{2In} + 3H_2F_2$$

A. Thiel, Z. anorg. Chem., **40**, 280 (1904) *28*

InF_3

H_2O **III-2353**

By boiling a solution of indium fluoride, indium trioxide is formed.

$$2InF_3 + 3H_2O \rightarrow \underline{In_2O_3} + 6HF$$

A. Thiel, Z. anorg. Chem., **40**, 280 (1904) *28*

$In(NO_3)_3$

$NaHSO_3$ **III-2354**

An insoluble basic sulfite is formed when a nearly neutral solution of indium salt is boiled with an excess of $NaHSO_3$.

$$4In(NO_3)_3 + 4NaHSO_3 + 10H_2O$$
$$\rightarrow In_2(SO_3)_3 \cdot 2In(OH)_3 \cdot 5H_2O + (4NaNO_3 + SO_2 + 8HNO_3)$$

Hillebrand and Lundell, Applied Inorganic Chemistry, p. 380 *93*

$In(OH)_3$

H_2SO_4 **III-2355**

An indium sulfate solution is formed when a slight excess of sulfuric acid is added to a suspension of indium hydroxide.

$$2In(OH)_3 + 3H_2SO_4 \rightarrow In_2(SO_4)_3 + 6H_2O$$

R. P. Seward, J. Am. Chem. Soc., **55**, 2740 (1933) *1*

$In(OH)_3$

H_2SeO_4 **III-2356**

Indium selenate is formed when indium hydroxide dissolves in selenic acid.

$$2In(OH)_3 + 3H_2SeO_4 + 4H_2O \rightarrow \underline{In_2(SeO_4)_3 \cdot 10H_2O}$$

F. C. Mathers and C. G. Schluederberg, J. Am. Chem. Soc., **30**, 214 (1908) *1*

$In(OH)_3$

III-2357

NaOH

$(NH_4)_2CO_3$

Indium hydroxide dissolves in an excess of sodium or potassium hydroxide but the solution becomes turbid on standing and is reprecipitated by boiling with ammonium carbonate.

(a) $In(OH)_3 + 3NaOH \rightarrow (Na_3InO_3 + 3H_2O)$

(b) $2Na_3InO_3 + 3(NH_4)_2CO_3 + 6H_2O \rightarrow \underline{2In(OH)_3} + (3Na_2CO_3 + 6NH_4OH)$

Treadwell and Hall, Analytical Chemistry, 5th. Ed., **1**, 539 *93*

In_2O_3

III-2358

C

Heating indium trioxide with carbon reduces the oxide to the metal.

$$2In_2O_3 + 3C \rightarrow 4In + (3CO_2)$$

Treadwell and Hall, Analytical Chemistry, 5th. Ed., **1**, 538 *93*

In_2O_3

III-2359

Cl_2

The heating of an intimately ground mixture of pure indium oxide and excess charcoal in a stream of dry chlorine and nitrogen will yield anhydrous indium trichloride.

$$(2In_2O_3 + 6Cl_2 \rightarrow 4InCl_3 + 3O_2)$$

T. Moeller, J. Am. Chem. Soc., **63**, 1206 (1941) *3*

In_2O_3

III-2360

Cl_2

By passing a current of chlorine over the heated oxide of indium, indium trichloride, which is very hygroscopic, is obtained.

$$2In_2O_3 + 6Cl_2 \rightarrow 4InCl_3 + 3O_2$$

T. Richter, Compt. rend., **64**, 828 (1867) *29*

In_2O_3

HCl **III-2361**

The ignited oxide dissolves slowly in cold acids, but readily in hot, dilute acids.

$$In_2O_3 + 6HCl \rightarrow (2InCl_3 + 3H_2O)$$

Treadwell and Hall, Analytical Chemistry, 5th. Ed., **1**, 538 *93*

In_2O_3

HF **III-2362**

By heating indium oxide gently with hydrofluoric acid, crystals of trihydrated indium fluoride are formed.

$$In_2O_3 + 6HF + 3H_2O \rightarrow \underline{2(InF_3 \cdot 3H_2O)}$$

A. Thiel, Z. anorg. Chem., **40**, 280 (1904) *28*

In_2O_3

H_2 **III-2363**

Indium is obtained by reducing the dried oxide with hydrogen in a porcelain crucible.

$$In_2O_3 + 3H_2 \rightarrow 2In + 3H_2O$$

T. Richter, Compt. rend., **64**, 828 (1867) *29*
93

In_2O_3

Mg **III-2364**

Magnesium reduces indium oxide with great violence when heated together in a current of hydrogen.

$$In_2O_3 + 3Mg \rightarrow 2In + 3MgO$$

H. F. Keller, J. Am. Chem. Soc., **16**, 839 (1894) *1*

In_2O_3 **III-2365**

ε

A solution of indium oxide in sulfuric acid with the addition of a small quantity of sodium citrate can be electrolized yielding metallic indium at the cathode.

(a) $In_2O_3 + 3H_2SO_4 \rightarrow In_2(SO_4)_3 + 3H_2O$

(b) $2In_2(SO_4)_3 + 6H_2O \rightarrow 4In + 3O_2 + 6H_2SO_4$

L. R. Westbrook, Can. Chem. & Met., **19**, 246 (1930) *15*

In_2S **III-2366**

Δ

On heating indium monosulfide in air, indium trioxide is formed.

$$In_2S + 5O \rightarrow \underline{In_2O_3} + SO_2$$

A. Thiel, Z. anorg. Chem., **40**, 280 (1904) *28*

In_2S_3 **III-2367**

H

By heating indium trisulfide in a stream of hydrogen, black colored indium monosulfide is formed.

$$In_2S_3 + 4H \rightarrow \underline{In_2S} + 2H_2S$$

A. Thiel, Z. anorg. Chem., **40**, 280 (1904) *28*

$In_2(SO_4)_3$ **III-2368**

C_2H_5OH

A basic indium sulfate is formed when alcohol is added to a solution of indium sulfate.

$$In_2(SO_4)_3 + 2C_2H_5OH + 5H_2O \rightarrow \underline{In_2O(SO_4)_2 \cdot 6H_2O} + (C_2H_5)_2SO_4$$

R. P. Seward, J. Am. Chem. Soc., **55**, 2742 (1933) *1*

$In_2(SO_4)_3$

H_2S **III-2369**

When hydrogen sulfide is passed into a dilute, weak acid (e.g., acetic acid) solution of indium sulfate, a yellow precipitate of indium sesquisulfide is thrown down.

$$In_2(SO_4)_3 + 3H_2S \rightarrow \underline{In_2S_3} + 3H_2SO_4$$

T. Richter, Compt. rend., **64**, 828 (1867) *29*

$In_2(SO_4)_3$

H_2SO_4 **III-2370**

Indium acid sulfate crystallizes when an excess of sulfuric acid is added to a solution of indium sulfate.

$$In_2(SO_4)_3 + H_2SO_4 + 7H_2O \rightarrow In_2(SO_4)_3 \cdot H_2SO_4 \cdot 7H_2O$$

R. P. Seward, J. Am. Chem. Soc., **55**, 2740 (1933) *1*

$In_2(SO_4)_3$

NH_4OH **III-2371**

Indium hydroxide, precipitated from indium sulfate solution by ammonia, is converted into the oxide by ignition.

(a) $$In_2(SO_4)_3 + 6NH_4OH \rightarrow \underline{2In(OH)_3} + 3(NH_4)_2SO_4$$

(b) $$2In(OH)_3 \rightarrow In_2O_3 + (3H_2O)$$

R. P. Seward, J. Am. Chem. Soc., **55**, 2740 (1933) *1*

$In_2(SO_4)_3$

$Na_2C_2O_4$ **III-2372**

Indium sulfate solution reacts with a solution of sodium oxalate to precipitate octahedral crystals according to equation (a). Potassium and ammonium oxalate react similarly.

(a) $$In_2(SO_4)_3 + 4Na_2C_2O_4 + 6H_2O \rightarrow 2Na[In(C_2O_4)_2(H_2O)_2] \cdot H_2O + 3Na_2SO_4)$$

(b) $$In_2(SO_4)_3 + 4K_2C_2O_4 + 6H_2O \rightarrow 2K[In(C_2O_4)_2(H_2O)_2] \cdot H_2O + 3K_2SO_4$$

(c) $$In_2(SO_4)_3 + 4(NH_4)_2C_2O_4 + 6H_2O \rightarrow 2NH_4[In(C_2O_4)_2(H_2O)_2] \cdot H_2O + 3(NH_4)_2SO_4$$

T. Moeller, J. Am. Chem. Soc., **62**, 2444 (1940) *3*

$In_2(SeO_4)_3 \cdot 10H_2O$ **III-2373**

Cs_2SeO_4

Beautiful colorless crystals of indium cesium selenate are formed when solutions of indium and cesium selenates are allowed to crystallize.

$$In_2(SeO_4)_3 \cdot 10H_2O + Cs_2SeO_4 \rightarrow 2CsIn(SeO_4)_2 \cdot 10H_2O$$

F. C. Mathers and C. G. Schluederberg, J. Am. Chem. Soc., **30**, 215 (1908) *1*

$[In_2(SeO_3)_3]_3 \cdot [In(OH)_3]_2 \cdot 22(H_2O)$ **III-2374**

SeO_2

H_2O

When the 3:2:22 hydrated basic indium selenite is digested with selenium dioxide in water at 60° hexa-hydrated normal indium selenite is formed as a crystalline powder.

$$[In_2(SeO_3)_3]_3 \cdot [In(OH)_3]_2 \cdot 22(H_2O) + 3SeO_2 + 3H_2O \rightarrow 4In_2(SeO_3)_3 \cdot 6(H_2O) + 4H_2O$$

L. F. Nilson, Nova Acta. Soc. Uppsal. [3], Vol. **9**, No. 7, 100 (1874) *10*

IODINE

$Ag_2H_3IO_6$ **III-2375**

Ag_2O

HNO_3

A hot dilute aqueous solution of secondary silver paraperiodate is treated with silver oxide. The solution is filtered and allowed to cool. A black crystalline powder precipitates. This is silver mesoperiodate.

(a) $$Ag_2O + 2HNO_3 \rightarrow 2AgNO_3 + H_2O$$

(b) $$Ag_2H_3IO_6 + AgNO_3 \rightarrow Ag_3IO_5 + HNO_3 + H_2O$$

A. Rosenheim and O. Liebknecht, Ann., **308**, 57 (1899) *20*

$Ag_2H_3IO_6$ **III-2376**

(H_2O)

Δ

Silver periodate heated in aqueous solution decomposes to form secondary silver mesoperiodate and water.

$$Ag_2H_3IO_6 \rightarrow Ag_2HIO_5 + H_2O$$

A. Rosenheim and O. Liebknecht, Ann., **308**, 55 (1899) *20*

$Ba(IO_3)_2$

H_2SO_4 **III-2377**

Barium iodate (prepared by reacting iodine with potassium chlorate and double decomposition of the potassium iodate with baryta) reacts with sulfuric acid to form iodic acid. The iodic acid thus obtained however, contains various reaction impurities, but may be purified by crystallization from nitric acid.

$$Ba(IO_3)_2 + H_2SO_4 \rightarrow \underline{BaSO_4} + 2HIO_3$$

M. Guichard, Compt. rend., **148**, 923 (1909) *38*
A. B. Lamb, W. C. Bray and W. J. Geldard, J. Am. Chem. Soc., **42**, 1636 (1920) *19*

CHI_3

AgCl **III-2378**

On heating gently and stirring to assure contact between reactants silver chloride and iodoform will react to produce silver iodide and chloroform. The iodoform is dissolved in a little alcohol.

$$3AgCl + CHI_3 \rightarrow 3AgI + CHCl_3$$

Oechsner de Coninck, Bull. soc. chim. (France), [(4)], **5**, 63 (1909) *51*

CHI_3

O_2 **III-2379**

The oxidation of iodoform by oxygen is accelerated by sunlight.

$$4CHI_3 + 5O_2 \rightarrow 6I_2 + 4CO_2 + 2H_2O$$

N. Dhar, J. Chem. Soc. (London), **111**, 756 (1907) *57*

CH_3I

$(CH_3)_4Fe(CN)_6$ **III-2380**

Tetramethyl ferrocyanide combines with methyl iodide at 100°C to form hexamethyl ferrocyanogen iodide.

$$2CH_3I + (CH_3)_4Fe(CN)_6 \rightarrow [(CH_3)_6Fe(CN)_6]I_2$$

E. G. J. Hartley, J. Chem. Soc. (London), **106**, 1304 (1916) *57*

CH_3I

III-2381

Zn

A baro-buret method of gas analysis is based on the following equation.

$$2CH_3I + 2Zn + 2H_2O \rightarrow ZnI_2 + Zn(OH)_2 + 2CH_4$$

H. S. Booth and N. C. Jones, Anal. Ed., **2**, 237 (1930) *44*

CNI

III-2382

HCl

When concentrated hydrochloric acid is reacted with cyanogen iodide at a temperature between 20–30°, hydrocyanic acid and iodine monochloride are formed.

$$CNI + HCl \rightarrow HCN + ICl$$

F. D. Chattaway and J. M. Wadmore, J. Chem. Soc. (London), **81**, 198 (1902) *102*

CNI

III-2383

HI

When a solution of cyanogen iodide is treated with hydrogen iodide, hydrocyanic acid and iodine are formed.

$$CNI + HI \rightarrow HCN + I_2$$

F. D. Chattaway and J. M. Wadmore, J. Chem. Soc. (London), **81**, 198 (1902) *102*

CNI

III-2384

KOH

(a) When a solution of cyanogen iodide is treated with potassium hydroxide, potassium cyanide, potassium hypoiodite and water are formed. (b) Part of the potassium hypoiodite escapes reduction by the potassium cyanide formed, however, some produces potassium cyanate and potassium iodide.

(a) $$CNI + 2KOH \rightarrow KCN + KOI + H_2O$$

(b) $$KCN + KOI \rightarrow KCNO + KI$$

F. D. Chattaway and J. M. Wadmore, J. Chem. Soc. (London), **81**, 199 (1902) *102*

C_2H_5I

$AgNO_2$ **III-2385**

The double decomposition of ethyl iodide and silver nitrite will yield silver iodide and ethyl nitrite.

$$C_2H_5I + AgNO_2 \rightarrow AgI + C_2H_5NO_2$$

V. Chiminello, Gazz. chim. ital., **25:2**, 411 (1895) *21*

C_2H_5I

$AgNO_2$ **III-2386**

$HgNO_2$

Silver nitrite reacts with ethyl iodide by metathesis. Mercurous nitrite reacts in a similar way.

(a) $AgNO_2 + C_2H_5I \rightarrow C_2H_5NO_2 + \underline{AgI}$

(b) $HgNO_2 + C_2H_5I \rightarrow C_2H_5NO_2 + \underline{HgI}$

P. Neogi, J. Chem. Soc. (London), **105**, 2372 (1914) *57*

C_2H_5I

$AgNO_3$ **III-2387**

Ethyl iodide reacts with silver nitrate at 0° temperature to yield silver iodide and ethyl nitrate:

$$C_2H_5I + AgNO_3 \rightarrow \underline{AgI} + C_2H_5NO_3$$

V. Chiminello, Gazz. chim. ital., **25²**, 416 (1895) *21*

C_2H_5I

Ag_2SO_4 **III-2388**

Under ordinary conditions the double decomposition of ethyl iodide and silver sulfate yield silver iodide and ethyl sulfate.

$$2C_2H_5I + Ag_2SO_4 \rightarrow \underline{2AgI} + (C_2H_5)_2SO_4$$

V. Chiminello, Gazz. chim. ital., **25²**, 411 (1895) *21*

C_2H_5I

III-2389

CH_3COONa

The double decomposition of ethyl iodide and sodium acetate will give sodium iodide and ethyl acetate.

$$C_2H_5I + CH_3COONa \rightarrow NaI + C_2H_5COOCH_3$$

V. Chiminello, Gazz. chim. ital., **25**², 411 (1895) *21*

C_2H_5I

III-2390

CH_3OH

Under ordinary conditions ethyl iodide and methyl alcohol will give methyl iodide and ethyl alcohol.

$$C_2H_5I + CH_3OH \rightarrow CH_3I + C_2H_5OH$$

V. Chiminello, Gazz. chim. ital., **25**², 417 (1895) *21*

C_2H_5I

III-2391

CH_3ONa

The double decomposition of ethyl iodide and sodium methylate is very rapid as represented below:

$$C_2H_5I + CH_3ONa \rightarrow NaI + C_2H_5OCH_3$$

V. Chiminello, Gazz. chim. ital., **25**², 411 (1895) *21*

C_2H_5I

III-2392

KCN

The double decomposition at ordinary temperature of ethyl iodide and potassium cyanide yields potassium iodide and ethyl cyanide.

$$C_2H_5I + KCN \rightarrow KI + C_2H_5CN$$

V. Chiminello, Gazz. chim. ital., **25**², 411 (1895) *21*

C_2H_5I

III-2393

$N(C_2H_5)_3$

Ethyl iodide dissolved in benzyl alcohol reacts vigorously with triethylamine.

$$C_2H_5I + N(C_2H_5)_3 \rightarrow N(C_2H_5)_4I$$

N. Menschutkin, Z. physik. Chem., **6**, 41 (1890) *25*

C_3H_5I

Zn **III-2394**

(Cu)

If allyl iodide be heated for two hours in a water bath, in the presence of a dry zinc—copper couple, zinc iodide is formed. Furthermore, four fifths of the organic product are di-allyl, (a), whereas the other one fifth seems to be an isomeric or polymeric form of allylene. (b) This latter compound seems to be formed by the reduction of the allylene by some zinc oxide impurities.

(a) $$2C_3H_5I + Zn \rightarrow ZnI_2 + C_6H_{10}$$

(b) $$(ZnO + C_6H_{10} \rightarrow Zn + 2C_3H_4 + H_2O)$$

J. H. Gladstone and A. Tribe, J. Chem. Soc. (London), **27**, 209 (1874) *48*

$(C_9H_6O_2)_4 \cdot KI \cdot I_3$

HCl **III-2395**

Coumarin-potassium iodide-periodide is treated at moderate temperatures with fuming hydrochloric acid. On cooling needles of bright green color, coumarin-hydrogen iodide-periodide, are formed.

$$(C_9H_6O_2)_4 \cdot KI \cdot I_3 + HCl \rightarrow \underline{(C_9H_6O_2)_4 \cdot HI \cdot I_3} + KCl$$

H. Simonis, Ber., **50:2**, 1137 (1917) *28*

FeI_2

H_2O_2 **III-2396**

Free iodine is produced by the action of hydrogen peroxide on ferrous iodide.

$$2FeI_2 + 3H_2O_2 \rightarrow 2Fe(OH)_3 + 2I_2$$

M. C. Weltzien, Chem. News, **13**, 159 (1866) *101*

HI

Ag **III-2397**

Silver reacts with liquid hydriodic acid with the evolution of hydrogen and formation of silver iodide.

$$2HI + 2Ag \rightarrow 2AgI + H_2$$

R. S. Norris and F. G. Cottrell, Am. Chem. J., **18**, 99 (1896) *1*

HI

III-2398

$CH_3C_6H_4SO_2 \cdot NH_2$

Fuming hydriodic acid will react with toluene sulfamide yielding iodine and ammonium iodide.

$$7HI + CH_3C_6H_4SO_2 \cdot NH_2 \rightarrow 6I + NH_4I + CH_3 \cdot C_6H_4 \cdot SH + 2H_2O$$

Emil Fischer, Ber., **48**, 93 (1915) *28*

HI

III-2399

$CaSO_4$

The alkaline earth sulfates, though not decomposed by cold concentrated hydriodic acid, are attacked at higher temperatures, the best results being obtained at a temperature just below the boiling point of the constant-boiling acid.

$$CaSO_4 + 10HI \rightarrow CaI_2 + 4I_2 + 4H_2O + H_2S$$

E. R. Caley and M. G. Burford, Anal. Ed., **8**, 63 (1936) *44*

HI

III-2400

$FeCl_3$

Iodine may be set free from hydriodic acid by the action of ferric chloride.

$$2HI + 2FeCl_3 \rightarrow I_2 + 2FeCl_2 + 2HCl$$

Meyerhoffer, Z. physik. Chem., **2**, 597 (1888)
Ref., Morris Loch, Am. Chem. J., **13**, 148 (1891) *17*

HI

III-2401

Fe_2O_3

Iodine may be liberated from iodides by ferric oxide.

$$2HI + Fe_2O_3 \rightarrow I_2 + 2FeO + H_2O$$

F. J. Hallinan and W. R. Thompson, J. Am. Chem. Soc., **61**, 268 (1939) *16*

HI

$HBrO_3$ **III-2402**

Under normal conditions hydriodic acid will reduce hydrobromic acid to give hydrogen bromide, iodine and water.

$$HBrO_3 + 6HI \rightarrow HBr + 3I_2 + 3H_2O$$

E. Oliveri Mandala, Gazz. chim. ital., **40**[1], 108 (1910) *21*

HI

HCN **III-2403**

By passing a current of dry hydrogen cyanide into a flask containing dry hydriodic acid, or by passing a current of dry hydrogen iodide into hydrogen cyanide at 0°, a reaction takes place in which the two compounds combine with ease, precipitating a white compound.

$$HI + HCN \rightarrow \underline{HI \cdot HCN}$$

A. Gautier, Compt. rend., 61, 380 (1865)
Ref., H. Gal, Compt. rend., 61, 644 (1865) *29*

HI

HIO_3 **III-2404**

A mixture of hydriodic acid and iodic acid solutions react slowly to liberate iodine.

$$5HI + HIO_3 \rightarrow 3I_2 + 3H_2O$$

H. S. Taylor, J. Chem. Soc. (London), **103**, 34 (1913) *57*

HI

HIO_3 **III-2405**

When hydrogen iodide and iodic acid react with each other, free iodine and water are produced.

$$5HI + HIO_3 \rightarrow 3I_2 + 3H_2O$$

E. B. R. Prideaux, J. Chem. Soc. (London), **89**, 316 (1906) *102*
G. Magnanini, Gazz. chim. ital., **20**, 379 (1890) *21*

HI

III-2406

HIO_3

HCl

In the volumetric determination of vanadium as vanadate, an excess of standard iodide is added to reduce the vanadium in 6 molal HCl solution. The iodide is converted to iodine monochloride by titration with iodate.

$$2HI + HIO_3 + 3HCl \rightarrow 3ICl + 3H_2O$$

E. H. Swift and R. H. Hoeppel, J. Am. Chem. Soc., **51**, 1366 (1929) *16*

HI

III-2407

HNO_2

Iodine may be liberated from iodides by nitrites.

$$2HI + 2HNO_2 \rightarrow I_2 + 2NO + 2H_2O$$

F. J. Hallinan and W. R. Thompson, J. Am. Chem. Soc., **61**, 268 (1939) *16*

HI

III-2408

HNO_2

The reaction between iodide and nitrous acid occurs only in acid medium.

$$HI + HNO_2 \rightarrow H_2O + NO + \tfrac{1}{2}I_2$$

C. A. Abeledo and I. M. Kolthoff, J. Am. Chem. Soc., **53**, 2893 (1931) *16*

HI

III-2409

HN_3

When conc. hydriodic acid (sp. g. 1.5) is mixed with a 1% solution of triazoic acid, iodine and nitrogen are evolved.

$$2HI + HN_3 \rightarrow NH_3 + N_2 + I_2$$

A. W. Browne and A. G. Hoel, J. Am. Chem. Soc., **44**, 2116 (1922) *16*

HI

H_2O_2 **III-2410**

Hydrogen peroxide added to an acid iodide solution is at first reduced (a). Conversely when hydrogen peroxide is added to an aqueous iodine solution it is oxidixed (b), so that the only measurable reaction (c) is the sum of (a) and (b).

(a) $$2HI + H_2O_2 \rightarrow I_2 + 2H_2O$$

(b) $$I_2 + H_2O_2 \rightarrow 2HI + O_2$$

(c) $$2H_2O_2 \rightarrow 2H_2O + O_2$$

H. A. Liebhafsky, J. Am. Chem. Soc., **56**, 2369 (1934) *16*

HI

MnO_2 **III-2411**

Iodine may be liberated from iodides by manganese dioxide.

$$2HI + MnO_2 \rightarrow I_2 + MnO + H_2O$$

F. J. Hallinan and W. R. Thompson, J. Am. Chem. Soc., **61**, 268 (1939) *16*

HI

O_2 **III-2412**

Hydriodic acid reacts with oxygen to produce water and free iodine.

$$4HI + O_2 \rightarrow 2H_2O + 2I_2$$

C. Weltzien, Chem. News, **13**, 160 (1866) *101*

HI

O_2 **III-2413**

The oxidation of hydriodic acid by oxygen is accelerated by sunlight.

$$4HI + O_2 \rightarrow 2H_2O + 2I_2$$

N. Dhar, J. Chem. Soc. (London), **111**, 756 (1917) *57*

HI

III-2414

SO_2

When sulfur dioxide is passed into an aqueous solution of hydriodic acid sulfur is deposited together with iodine.

$$SO_2 + 4HI \rightarrow S + 2H_2O + 4I$$

J. Volhard, Ann., **242**, 105 (1887) *20*

HI

III-2415

α-Amino

β-γ-dihydroxy butyric acid

P

If a mixture of α-amino, β-γ-dihydroxy *n*-butyric acid, hydriodic acid and red phosphorus is heated for 4–5 hours at 120°–125°C, α-amino-n-butyric acid is formed.

$$CH_2OHCHOHCHNH_2 \cdot COOH + 4HI + P_2 \rightarrow P_2I_4 + C_3H_6NH_2COOH + 2H_2O$$

E. Klenk and W. Diebold, Z. physiol. Chem., **198**, 25 (1931) *14*

HI·HCN

III-2416

H_2O

Water decomposes the hydrogen iodide-hydrogen cyanide compound, forming hydrogen iodide and ammonium formate.

$$HI \cdot HCN + 2H_2O \rightarrow HI + HCOONH_4$$

H. Gal, Compt. rend., **61**, 644 (1865) *29*

HI·HCN

III-2417

KOH

If potassium hydroxide in absolute alcohol is passed into the cooled alcoholic solution of the hydrogen iodide-hydrogen cyanide compound, potassium iodide precipitates immediately.

$$HI \cdot HCN + KOH \rightarrow \underline{KI} + NH_2COH$$

A. Gautier, Compt. rend., **61**, 381 (1865) *29*

HIO_2

SO_2 **III-2418**

Iodous acid is reduced by sulfur dioxide.

$$HIO_2 + SO_2 \rightarrow HIO + SO_3$$

H. Landolt, Sitzber, Akad. Bodin., **249**, (1885) *25*

HIO_3

CO **III-2419**

At a temperature of 150°C, carbon monoxide is oxidized to carbon dioxide by iodic acid.

$$2HIO_3 + 5CO \rightarrow I_2 + 5CO_2 + H_2O$$

A. H. Gill and E. H. Bartlett, J. Ind. Eng. Chem., **2**, 9 (1910) *22*

HIO_3

HCl **III-2420**

Iodic acid is reduced by hydrochloric when a solution containing the two substances is evaporated.

$$2HIO_3 + 10HCl \rightarrow I_2 + 5Cl_2 + 6H_2O$$

A. B. Lamb, W. C. Bray, and W. J. Geldard, J. Am. Chem. Soc., **42**, 1640 (1920) *1*
Ref., Sodini, Ber., **9**, 1126 (1876) *16*

HIO_3

HI **III-2421**

The equilibrium of the reaction given below is determined by EMF measurements.

$$HIO_3 + 5HI \rightleftharpoons 3I_2 + 3H_2O$$

R. Luther and G. V. Sammet, Z. Elektrochem., **11**, 293 (1905) *86*

HIO_3

$H_2C_2O_4$ **III-2422**

Iodine is obtained when oxalic acid reacts with iodic acid.

$$2HIO_3 + 5H_2C_2O_4 \rightarrow I_2 + 10CO_2 + 6H_2O$$

L. Rosenthaler, Z. anal. Chem., **61**, 221 (1922) *28*

HIO_3

III-2423

$H_2C_2O_4 \cdot 2H_2O$

H_2SO_4

Iodic acid is treated with oxalic acid in a 12% sulfuric acid solution to give carbon dioxide and iodine.

$$2HIO_3 + 5H_2C_2O_4 \rightarrow 10CO_2 + 6H_2O + I_2$$

L. Debourdeaux, Ann. chim. anal., **9**, 167 (1904) *76*

HIO_3

III-2424

H_2O_2

Hydrogen peroxide reduces iodic acid to free iodine.

$$5H_2O_2 + 2HIO_3 \rightarrow 5O_2 + I_2 + 6H_2O$$

W. C. Bray, J. Am. Chem. Soc., **43**, 1262 (1921) *1*

HIO_3

III-2425

H_2SO_3

A kinetic study is made of a series of simultaneous reactions, which proceed with various rates and are known as the "Landolt Reaction":

(a) $$HIO_3 + 3H_2SO_3 \rightarrow HI + 3H_2SO_4$$

(b) $$HIO_3 + 5HI \rightarrow 3I_2 + 3H_2O$$

(c) $$I_2 + H_2SO_3 + H_2O \rightarrow 2HI + H_2SO_4.$$

J. Eggert, Z. Elektrochem., **23**, 8 (1917) *86*

HIO_3

III-2426

H_2SO_4

A mixture of 60 g. powdered iodic acid and 200 g. concentrated sulfuric acid is heated for about 10–15 minutes. The product is kept over sulfuric acid for 5–6 days. Iodine dioxide separates as a yellow crystalline crust.

$$4HIO_3 \rightarrow 2I_2O_4 + 2H_2O + O_2$$

M. M. P. Muir, J. Chem. Soc. (London), **95**, 656 (1909). *57*
Ref., J. L. Howe, J. Am. Chem. Soc., **31**, 1293 (1909) *1*

HIO_3

$N_2H_4 \cdot H_2SO_4$ **III-2427**

A solution of iodic acid is added drop by drop to a solution of hydrazine sulfate. The reduction yields hydriodic acid immediately.

(a) $4HIO_3 + 5(N_2H_4 \cdot H_2SO_4) \rightarrow 5N_2 + 12H_2O + 5H_2SO_4 + 4I$

(b) $4I + N_2H_4 \rightarrow 4HI + N_2$

M. Schlatter, Z. anorg. Chem., **38**, 184 (1904) *28*

HIO_3

$Na_2S_2O_3$ **III-2428**

Iodine is liberated when sodium thiosulfate is titrated with an excess of iodic acid.

(a) $6Na_2S_2O_3 + 6HIO_3 \rightarrow 3Na_2S_4O_6 + 5NaIO_3 + NaI + 3H_2O$

(b) $5NaI + 6HIO_3 \rightarrow 5NaIO_3 + 3H_2O + 3I_2$

C. F. Walker, Am. J. Sci., **154**, 235
Ref., ibid, J. Am. Chem. Soc., **19**, 160 (1897) *1*

HIO_3

PbO_2 **III-2429**

Iodic acid and lead dioxide react to form periodic acid.

$$PbO_2 + 3IO_3^- + 2H^+ \rightarrow Pb(IO_3)_2 + IO_4^- + H_2O$$

E. Müller, Z. Elektrochem., **10**, 63 (1904) *86*

HIO_4

$CH_3 \cdot CHOH \cdot CHOH \cdot CH_3$ **III-2430**

2, 3-butylene glycol may be estimated by periodic acid according to the following reactions.

(a) $CH_3 \cdot CHOH \cdot CHOH \cdot CH_3 + HIO_4$
$\rightarrow 2CH_3CHO + HIO_3 + H_2O$

(b) $2CH_3CHO + 2NH_2OH \cdot HCl$
$\rightarrow 2CH_3CH{:}NOH + 2H_2O + 2HCl$

M. C. Brockmann and C. H. Werkman, Anal. Ed., **5**, 206 (1933) *44*

HIO_4

III-2431

Cs_2CO_3

Periodic acid is neutralized with cesium carbonate to prepare cesium periodate.

$$2HIO_4 + Cs_2CO_3 \rightarrow 2CsIO_4 + (H_2O + CO_2).$$

Wells, Am. Chem. J., **26**, 278 (1901) *57*
Ref., T. V. Barker, J. Chem. Soc. (London), **93**, 17 (1908) *102*

HIO_4

III-2432

HCl

Periodic acid oxidizes hydrochloric acid to chlorine.

$$2HCl + HIO_4 \rightarrow Cl_2 + HIO_3 + H_2O$$

H. H. Willard and L. H. Greathouse, J. Am. Chem. Soc., **39**, 2371 (1917) *1*

HIO_4

III-2433

H_2SO_3

Periodic acid in varying proportions, oxidizes sulfurous acid.

(a) $HIO_4 + 4H_2SO_3 \rightarrow HI + 4H_2SO_4$ (equivalent quantities)

(b) $HIO_4 + H_2SO_3 \rightarrow HIO_3 + H_2SO_4$ (excess of HIO_4)

Selmous, Ber., **21**, 230 (1888) *25*

HIO_4

III-2434

NH_4OH

When periodic acid is neutralized with aqueous ammonia, ammonium periodate precipitates.

$$HIO_4 + NH_4OH \rightarrow \underline{NH_4IO_4} + (H_2O)$$

T. V. Barker, J. Chem. Soc. (London), **93**, 17 (1908) *57* *102*

HOI

(NaOI) **III-2435**

Hypoiodites in presence of hypoiodous acid are converted to iodate and hydriodic acid. This action is accelerated by increase of temperature and concentration.

$$2HOI + (NaOI) \rightarrow (NaIO_3) + 2HI$$

H. S. Taylor, J. Chem. Soc. (London), **103**, 32 (1913) *67*

$H_2I_2O_6$

H_2 **III-2436**

ε

A solution of iodic acid electrolyzed between platinum electrodes at a potential of less than 0.8 volts deposits iodine on the cathode.

$$5H_2 + H_2I_2O_6 \rightarrow I_2 + 6H_2O$$

C. J. Reed, Trans. Am. Electrochem. Soc., **1**, 78 (1902) *8*

I

BaO_2 **III-2437**

When barium peroxide and excess iodine are ground together until the mixture is black, and then heated in a retort, barium iodide and iodine pentoxide are obtained.

$$5BaO_2 + 7I_2 \rightarrow 2I_2O_5 + 5BaI_2$$

Sementini, Ann., **4**, 170 (1832) *20*

I

CH_3COCHO **III-2438**

Iodine dissolved in potassium iodide will react with methyl glyoxal yielding sodium oxalate and iodoform.

$$4I_2 + CH_3COCHO + 7NaOH \rightarrow Na_2C_2O_4 + CHI_3 + 5NaI + 5H_2O$$

F. Fischler and R. Boettner, **74**, 30 (1928) *28*

I

III-2439

$C_9H_6O_2$

KBr

3 grams of coumarin, 3 grams of iodine and 15 grams of potassium bromide are completely dissolved. After digestion at higher temperatures the solution is cooled. The residue after drying consists of gold-greenish colored needles of coumarin-potassium bromide-periodide.

$$3I + KBr + 4C_9H_6O_2 \rightarrow \underline{(C_9H_6O_2)_4 \cdot KBr \cdot I_3}$$

28

H. Simonis, Ber., **50:2**, 1137 (1917)

I

III-2440

$C_9H_6O_2$

KI

3 grams of coumarin are disolved in ½ liter of boiling water which contains KI. To this solution is added a solution of 3 grams of iodine in 10 grams of potassium iodide. On heating (on the water bath) a dark colored liquid is formed from which, on cooling, dark green colored needles of coumarin-potassium iodide-periodide are obtained. The same type reaction occurs with sodium iodide.

(a) $3I + KI + 4C_9H_6O_2 + H_2O \rightarrow \underline{(C_9H_6O_2)_4 \cdot KI \cdot I_3} + H_2O$

(b) $3I + NaI + 4C_9H_6O_2 \rightarrow \underline{(C_9H_6O_2)_4 \cdot NaI \cdot I_3}$

28

H. Simonis, Ber., **50:2**, 1137 (1917)

I

III-2441

$C_9H_6O_2$

LiI

3 grams of coumarin are dissolved in ½ liter of boiling water which contains lithium iodide. To this solution is added a solution of 3 grams of iodine in 10 grams of lithium iodide. On heating (on the water bath) a dark colored liquid is formed from which on cooling bluish colored needles of coumarin-lithium iodide-periodide are obtained. The same type reaction occurs with rubidium iodide and cesium iodide.

(a) $3I + LiI + 4C_9H_6O_2 \rightarrow \underline{(C_9H_6O_2)_4 \cdot LiI \cdot I_3}$

(b) $3I + RbI + 4C_9H_6O_2 + H_2O \rightarrow \underline{(C_9H_6O_2)_4 \cdot RbI \cdot I_3 \cdot H_2O}$

(c) $3I + CsI + 4C_9H_6O_2 + H_2O \rightarrow \underline{(C_9H_6O_2)_4 \cdot CsI \cdot I_3 \cdot H_2O}$

28

H. Simonis, Ber., **50:2**, 1137 (1917)

I

KI **III-2442**

$C_{10}H_8O_2$

A solution of iodine in potassium iodide is added to a solution of 3-methylcoumarin in alcohol. Spinach-green needles of 3-methylcoumarin-potassium iodide-periodide are obtained. The same type reaction occurs with 4,7-dimethylcoumarin.

$$3I + KI + 4C_{10}H_8O_2 + H_2O \rightarrow \underline{(C_{10}H_8O_2)_4 \cdot KI \cdot I_3 \cdot H_2O}$$

H. Simonis, Ber., **50**:2, 1137 (1917) *28*

I

CaI_2 **III-2443**

By fusing together iodine with calcium iodide whereby an excess of iodine is necessary, calcium hexaiodide, calcium octaiodide, and calcium decaiodide in form of a crystalline mass are obtained. The same type reaction occurs with barium iodide and strontium iodide yielding the same classes of iodides.

(a) $CaI_2 + 4I \rightarrow CaI_6$

(b) $CaI_2 + 6I \rightarrow CaI_8$

(c) $CaI_2 + 8I \rightarrow CaI_{10}$

J. Meyer, Z. anorg. Chem., **30**, 113 (1902) *28*

I

CaI_2 **III-2444**

BaI_2

SrI_2

Mix powdered iodine with powdered calcium iodide. Heat to 70–80°. The mass liquefies. During the cooling period a crystalline mass of calcium tetraiodide is formed. The same type reaction occurs with barium and strontium iodides.

(a) $2I + CaI_2 \rightarrow \underline{CaI_4}$

(b) $2I + BaI_2 \rightarrow \underline{BaI_4}$

(c) $2I + SrI_2 \rightarrow \underline{SrI_4}$

J. Meyer, Z. anorg. Chem., **30**, 113 (1902) *28*

I

III-2445

H_2S

If a hydrogen sulfide solution is titrated with iodine the products are sulfur and hydriodic acid.

$$H_2S + I_2 \rightarrow S + 2HI$$

A. R. Jayson and R. E. Oesper, J. Ind. Eng. Chem., **9**, 975 (1917) *22*

I

III-2446

H_3PO_2

Hydriodic acid is formed when iodine and water are heated to 50°C, and 50% hypophosphorous acid added portionwise. The HI is distilled off as a constant boiling mixture.

$$(I_2 + H_3PO_2 + H_2O \rightarrow 2HI + H_3PO_3)$$

E. P. Clark, Anal. Ed., **10**, 677 (1938) *114*

I

III-2447

O_2

When iodine is heated in a tube with air or oxygen to a high temperature, iodine pentoxide is obtained.

$$4I + 5O_2 \rightarrow 2I_2O_5$$

Sementini, Ann., **4**, 170 (1832) *20*

I

III-2448

$Rb_2S_2O_3$

Iodine oxidizes rubidium thiosulfate to the tetrathionate.
A similar reaction occurs with cesium thiosulfate.

(a) $$2Rb_2S_2O_3 + I_2 \rightarrow Rb_2S_4O_6 + (2RbI)$$

(b) $$2Cs_2S_2O_3 + I_2 \rightarrow Cs_2S_4O_6 + (2CsI)$$

Meyer and Eggeling, Ber., **40**, 1351 (1907)
Ref., J. E. MacKenzie and H. Marshall, J. Chem. Soc. (London), **93**, 1730 (1908) *57*

I

3-nitrophenyl acetic acid **III-2449**

p-Nitrophenyl acetic acid was iodized in the 2 position by dissolving in glacial acetic acid, adding iodine and concentrated nitric acid dropwise during refluxing until there was no further evolution of oxides of nitrogen or evidence of iodine vapors.

$$I_2 + NO_2C_6H_4CH_2COOH \rightarrow HI + NO_2C_6H_3ICH_2COOH$$

G. Feasley, et. al., J. Am. Pharm. Assn., **30**, 41 (1941) *112*

IBr

ε **III-2450**

The electrolysis of iodine bromide in aqueous solution containing starch shows that the iodine goes to the cathode.

$$IBr \rightarrow I^+ + Br^-$$

de la Rive, Phil. Mag. [(2)], **3**, 145 (1828)
Ref., R. H. Clark and H. R. L. Streight, Trans. Roy. Soc. (Canada) [III], **22**, 323 (1928) *69*

ICN

HCl **III-2451**

The action of hydrogen halides on iodocyanogen shows that the iodine is chemically positive.

$$ICN + HCl \rightarrow HCN + ICl$$

F. D. Chattaway and J. M. Wadmore, J. Chem. Soc. (London), **81**, 195 (1902)
Ref., R. H. Clark and H. R. L. Streight, Trans. Roy. Soc. (Canada) [III], **23**, 77 (1929) *69*

ICN

HI **III-2452**

Iodine cyanide decomposes in the presence of an excess of hydriodic acid to form iodine and hydrogen cyanide.

$$ICN + HI \rightarrow I_2 + HCN$$

K. Seubert and W. Pollard, Ber., **23**, 1062 (1890)
Ref., L. H. F., J. Am. Chem. Soc., **12**, 225 (1890) *1*

ICN

III-2453

H_2O

Iodine chloride and bromide contain positive iodine and both give, on hydrolysis, hypoiodous acid which is unstable and undergoes an oxidation-reduction change to iodic acid and free iodine.

$$(5ICl + 3H_2O \rightarrow 5HCl + 2I_2 + HIO_3)$$

K. J. P. Orton and W. L. Blackmann, J. Chem. Soc. (London), **77**, 830 (1900)
Ref., R. H. Clark and H. R. L. Streight, Trans. Roy. Soc. (Canada) [III], **22**, 323 (1928) *69*

ICN

III-2454

KOH

On boiling with potash, iodocyanogen forms potassium iodate.

(a) $$ICN + 2KOH \rightarrow KCN + KOI + H_2O$$

(b) $$3KOI \rightarrow KIO_3 + 2KI$$

J. U. Nef, Ann., **287**, 265 (1895)
Ref., R. H. Clark and H. R. L. Streight, Trans. Roy. Soc. (Canada), [III], **22**, 323 (1928) *69*

ICN

III-2455

ε

In the electrolysis of iodocyanogen between silver electrodes, the iodine travels to the anode, except in pyridine solution, when it goes to the cathode. Iodocyanogen thus behaves as an electromer.

$$ICN \rightarrow \overset{+}{I}\overset{-}{C}N \rightarrow \overset{-}{I}\overset{+}{C}N$$

R. H. Clark and H. R. L. Streight, Trans. Roy. Soc. (Canada) [III], **22**, 323 (1928) *69*

ICl

III-2456

AgCN

Iodine monochloride reacts vigorously with silver cyanide.

$$ICl + AgCN \rightarrow \underline{AgCl} + ICN$$

J. Cornog, H. W. Horrabin and R. A. Karges, J. Am. Chem. Soc., **60**, 430 (1938) *16*

ICl

AgCNS **III-2457**

With silver thiocyanate, iodine monochloride forms iodine trithiocyanate.

$$3ICl + 3AgCNS \rightarrow 3AgCl + I(CNS)_3 + I_2$$

J. Cornog, H. W. Horrabin and R. A. Karges, J. Am. Chem. Soc., **60**, 430 (1938) *16*

ICl

AgCl **III-2458**

ε

Faraday electrolyzed anhydrous iodine chloride in molten silver chloride and found iodine and silver at the cathode.

$$(ICl + AgCl \rightarrow I^+ + Ag^+ + Cl_2^-)$$

Faraday, "Ostwald's Klassiker," **86**, 43
Ref., R. H. Clark and H. R. L. Streight, Trans. Roy. Soc. (Canada) [III], **22**, 323 (1928) *69*

ICl

CH_3NH_2 **III-2459**

After a considerable quantity of iodine monochloride has been added to methyl amine, a yellow precipitate of methyl-diiodoamine is formed.

$$2ICl + CH_3NH_2 \rightarrow \underline{CH_3NI_2} + 2HCl$$

K. J. P. Orton and W. L. Blackman, J. Chem. Soc. (London), **77**, 833 (1900) *102*

ICl

$Cd(CN)_2$ **III-2460**

Iodine monochloride reacts vigorously with cadmium cyanide.

$$2ICl + Cd(CN)_2 \rightarrow CdCl_2 + 2ICN$$

J. Cornog, H. W. Horrabin, and R. A. Karges, J. Am. Chem. Soc., **60**, 430 (1938) *16*

ICl

Cl_2 **III-2461**

H_2O

The decomposition of iodine chloride is studied by potentiometric titration:

(a) $$IO_3^- + 6H^+ + Cl^- + 6(-) \rightarrow ICl + 3H_2O$$

(b) $$I^- + Cl^- + 2(+) \rightarrow ICl$$

(c) $$ICl + 2Cl_2 + 3H_2O \rightarrow HIO_3 + 5H^+ + 5Cl^-$$

E. Müller and D. Junck, Z. Elektrochem., **31**, 209 (1925) *86*

ICl

III-2462

CsCN

Iodine monochloride reacts vigorously with cesium cyanide.

$$ICl + CsCN \rightarrow CsCl + ICN$$

J. Cornog, H. W. Horrabin, and R. A. Karges, J. Am. Chem. Soc., **60**, 430 (1938) *16*

ICl

III-2463

CuCN

Iodine monochloride reacts vigorously with cuprous cyanide.

$$ICl + CuCN \rightarrow CuCl + ICN$$

J. Cornog, H. W. Horrabin and R. A. Karges, J. Am. Chem. Soc., **60**, 430 (1938) *16*

ICl

III-2464

Cu_2C_2

Diiodo acetylene is obtained when iodine monochloride reacts with copper acetylide.

$$2ICl + Cu_2C_2 \rightarrow C_2I_2 + Cu_2Cl_2$$

L. B. Howell and W. A. Noyes, J. Am. Chem. Soc., **42**, 993 (1920) *1*

ICl

III-2465

H_2O

Iodine mono chloride hydrolyzes forming hypoiodous acid and hydrochloric acid.

$$ICl + H_2O \rightarrow HIO + HCl$$

L. W. Andrews, J. Am. Chem. Soc., **25**, 757 (1903) *1*

ICl

H_2 **III-2466**

Gaseous iodine monochloride and hydrogen react to give free gaseous iodine and hydrogen chloride.

$$2ICl + H_2 \rightarrow I_2 + 2HCl$$

A. Sherman and N. Li., J. Am. Chem. Soc., **58**, 690 (1936) *3*

ICl

H_2O **III-2467**

Iodine monochloride is partly hydrolized in aqueous solution; hypoiodous acid and hydrochloric acid are formed (a). The hypoiodous acid subsequently decomposes into iodine, iodic acid and water (b). Other products of the decomposition of iodine chloride are iodine trichloride and a hydrogen chloride-iodine monochloride addition compound.

(a) $$ICl + H_2O \rightarrow IOH + HCl$$

(b) $$5IOH \rightarrow 2I_2 + HIO_3 + 2H_2O$$

V. Cofman, J. Chem. Soc. (London), **115**:**2**, 1042 (1919) *48*

ICl

KCN **III-2468**

Iodine monochloride reacts vigorously with potassium cyanide.

$$ICl + KCN \rightarrow KCl + ICN$$

J. Cornog, H. W. Horrabin, and R. A. Karges, J. Am. Chem. Soc., **60**, 430 (1938) *16*

ICl

KCNO **III-2469**

Iodine monochloride reacts vigorously with potassium cyanate.

$$3ICl + 3KCNO \rightarrow 3KCl + I(CNO)_3 + I_2$$

J. Cornog, H. W. Horrabin and R. A. Karges, J. Am. Chem. Soc., **60**, 429 (1938) *16*

ICl

III-2470

KCNS

Iodine monochloride reacts with a thiocyanate. The mixture of the chloride and thiocyanate are placed in a glass stoppered flask and cooled by an ice water bath for several hours then water is added. Any free iodine is removed with carbon tetrachloride. A cyanide and a cyanate react similarly with iodine monochloride.

(a) $$3KCNS + 3ICl \rightarrow 3KCl + I(CNS)_3 + I_2$$

(b) $$KCN + ICl \rightarrow KCl + ICN$$

(c) $$3KCNO + 3ICl \rightarrow 3KCl + I(CNO)_3 + I_2$$

J. Cornog, H. W. Horrabin and R. A. Karges, J. Am. Chem. Soc., **60**, 429 (1938) *3*
16

ICl

III-2471

KCl

Potassium chloride in excess is placed in iodine monochloride at 50°; the resulting mixture is kept in a thermostat at 40° for two or more hours, and the material remaining undissolved is removed by filtration, the excess iodine monochloride is removed by evaporation. Ammonium, rubidium, and cesium chloride react similarly.

(a) $$KCl + ICl \rightarrow KCl \cdot ICl$$

(b) $$NH_4Cl + ICl \rightarrow NH_4Cl \cdot ICl$$

(c) $$RbCl + ICl \rightarrow RbCl \cdot ICl$$

(d) $$CsCl + ICl \rightarrow CsCl \cdot ICl$$

J. Cornog, H. W. Horrabin, and R. A. Karges, J. Am. Chem. Soc., **60**, 429 (1938) *3*

ICl

III-2472

KI

Potassium iodide decomposes iodine monochloride with the formation of free iodine.

$$ICl + KI \rightarrow I_2 + KCl$$

Hennecke, Pharm. Ztg., **49**, 957
Ref., J. Am. Chem. Soc., **27**, 1347 (1905) *1*

ICl

KOH **III-2473**

If iodine monochloride is treated with an alkali, a hypoiodite, a chloride and water result.

$$6KOH + 3ICl \rightarrow 3KIO + 3KCl + 3H_2O$$

J. K. P. Orton and W. L. Blackman, J. Chem. Soc. (London), **77**:2, 830 (1900) *103*

ICl

KOH **III-2474**

Iodine monochloride reacts with potassium hydroxide.

$$5ICl + 6KOH \rightarrow KIO_3 + 2I_2 + 3H_2O + 5KCl$$

Philip, Ber., **3**, 4 (1870) 25

ICl

NH_3 **III-2475**

Nitrogen iodide forms when strong ammonia is poured over a mixture of crushed ice and iodine monochloride solution.

$$2NH_3 + 3ICl \rightarrow N_2H_3I_3 + (3HCl)$$

F. D. Chattaway and K. J. P. Orton, Am. Chem. J., **23**, 364 (1900) *1*

ICl

NH_3 **III-2476**

NaOH

If iodine chloride is formed in the presence of ammonia and sodium hydroxide the reaction tends to produce sodium chloride, nitrogen triiodide and water.

$$3ICl + NH_3 + 3NaOH \rightarrow 3NaCl + NI_3 + 3H_2O$$

Trillat and Turchet, Ann. chim. anal., **10**, 179 (1905) *76*

ICl

NH_4CN **III-2477**

Ammonium cyanide reacts vigorously with iodine monochloride.

$$ICl + NH_4CN \rightarrow NH_4Cl + ICN$$

J. Cornog, H. W. Horrabin and R. A. Karges, J. Am. Chem. Soc., **60**, 429 (1938) *16*

ICl

III-2478

NH_4CNS

With ammonium thiocyanate iodine monochloride forms iodine trithiocyanate.

$$3ICl + 3NH_4CNS \rightarrow 3NH_4Cl + I(CNS)_3 + I_2$$

J. Cornog, H. W. Horrabin and R. A. Karges, J. Am. Chem. Soc., **60**, 430 (1938) *16*

ICl

III-2479

$Pb(CN)_2$

Iodine monochloride reacts vigorously with lead cyanide.

$$2ICl + Pb(CN)_2 \rightarrow PbCl_2 + 2ICN$$

J. Cornog, H. W. Horrabin, and R. A. Karges, J. Am. Chem. Soc., **60**, 430 (1938) *16*

ICl

III-2480

$Pb(CNO)_2$

With lead cyanate, iodine monochloride forms iodine tricyanate.

$$6ICl + 3Pb(CNO)_2 \rightarrow 3PbCl_2 + 2I(CNO)_3 + 2I_2$$

J. Cornog, H. W. Horrabin and R. A. Karges, J. Am. Chem. Soc., **60**, 430 (1938) *16*

ICl

III-2481

$Pb(CNS)_2$

With lead thiocyanate, iodine monochloride forms iodine trithiocyanate.

$$6ICl + 3Pb(CNS)_2 \rightarrow 3PbCl_2 + 2I(CNS)_3 + 2I_2$$

J. Cornog, H. W. Horrabin, and R. A. Karges, J. Am. Chem. Soc., **60**, 430 (1938) *16*

ICl

III-2482

S_2Br_2

Iodine monochloride reacts with sulfur monobromide.

$$2ICl + S_2Br_2 \rightarrow 2IBr + S_2Cl_2$$

J. B. Hannay, J. Chem. Soc. (London), **26**, 823 (1873) *25*

ICl

Dehydrocholic acid **III-2483**

If iodine monochloride is allowed to react several days in glacial acetic acid with dehydrocholic acid at room temperature, diiodo dehydrocholic acid precipitates.

$$(2ICl + C_{24}H_{34}O_5 \rightarrow 2HCl + \underline{C_{24}H_{32}O_5I_2})$$

W. Borsche and K. Diacont, Z. physiol. Chem., **198**, 115 (1931) *14*

ICl

Δ **III-2484**

Iodine monochloride heated to 101° dissociates into iodine, and iodine trichloride.

$$3ICl \rightarrow I_2 + ICl_3$$

G. Oddo and E. Serra, Gazz. chim. ital., **29**[2], 352 (1899) *21*

ICl_3

CS_2 **III-2485**

Iodine trichloride reacts with carbon disulfide.

$$6ICl_3 + 4CS_2 \rightarrow 2CCl_4 + 2CSCl_2 + 3S_2Cl_2 + 3I_2$$

J. B. Hannay, Chem. News, **37**, 224 (1887) *25*

ICl_3

Cl_2O **III-2486**

Iodine trichloride reacts with chlorine monoxide.

$$2ICl_3 + 5Cl_2O \rightarrow I_2O_5 + 8Cl_2$$

H. Basset and E. Fielding, Chem. News, **54**, 205 (1886) *25*

ICl_3

H_2O **III-2487**

Iodine trichloride reacts with water.

$$2ICl_3 + 3H_2O \rightarrow HIO_3 + 5HCl + ICl$$

P. Schutzenberger, Compt. rend., **84**, 389 (1877) *25*

ICl_3

III-2488

Decomp.

Iodine trichloride decomposes.

$$ICl_3 \rightarrow ICl + Cl_2$$

J. Philipp, Ber., **3**, 4, (1870) *25*

IOH

III-2489

C_6H_5OH

Hypoiodous acid reacts with phenol; the corresponding iodophenol is formed with liberation of water.

$$IOH + C_6H_5OH \rightarrow C_6H_4I \cdot OH + H_2O$$

Selivanov, Ber., **27**, 1012 (1894)
Ref., V. Cofman, J. Chem. Soc. (London), **115**:2, 1048 (1919) *48*

$I(OH)_3$

III-2490

Decomp.

Iodine hydroxide decomposes spontaneously.

$$3I(OH)_3 \rightarrow 2HIO_3 + HI + 3H_2O$$

F. Fichter and Rohner, Ber., **42**, 4093 (1909) *25*

IO_3^-

III-2491

$Fe(CN)_6^{---}$

A reaction scheme with three simultaneous reactions, similar to the Landolt reaction is investigated and described:

(a) $IO_3^- + 6Fe(CN)_6^{---} + 6H^+ \rightarrow I^- + 6Fe(CN)_6^{----} + 3H_2O$

(b) $IO_3^- + 5I^- + 6H^+ \rightarrow 3I_2 + 3H_2O$

(c) $I_2 + 2Fe(CN)_6^{----} \rightleftharpoons 2I^- + 2Fe(CN)_6^{---}$

J. Eggert and B. Scharnow, Z. Elektrochem., **27**, 458 (1921) *86*

IO_3^-

III-2492

ε

Iodate ion is anodically oxidized in alkaline solution to periodate ion.

$$IO_3^- + 2OH^- + 2\oplus \rightarrow IO_4^- + H_2O$$

E. Müller, Z. Elektrochem., **10**, 57, 58 (1904) *86*

IO_4^-

H^+

I^- **III-2493**

The reduction of periodate (a) and iodate ion (b) with iodide ion is followed potentiometrically:

(a) $IO_4^- + 8H^+ + 7I^- \rightarrow 4I_2 + 4H_2O$

(b) $IO_3^- + 6H^+ + 5I^- \rightarrow 3I_2 + 3H_2O$

E. Müller and D. Junck, Z. Elektrochem., **31**, 200, 204 (1925) *86*

IO_4^-

I^- **III-2494**

The reaction between periodates and iodides is catalyzed by the action of hydrogen ions, light and platinum.

$$3IO_4^- + I^- \rightarrow 4IO_3^-$$

E. Müller, Z. Elektrochem., **9**, 707 (1903) *86*

I_2

AgCNO **III-2495**

Silver cyanate and iodine react slowly in cold water and rapidly when heated, to evolve carbon dioxide and form cyanic acid, which slowly decomposes to carbamide.

(a) $6AgCNO + 3I_2 + 3H_2O \rightarrow \underline{5AgI} + AgIO_3 + 6HCNO$

(b) $2HCNO + H_2O \rightarrow CO(NH_2)_2 + CO_2$

C. W. B. Normand, J. Chem. Soc. (London), **101**, 1857 (1912) *57*

I_2

AgCNS **III-2496**

When silver thiocyanate is shaken with iodine in water, hot or cold, a rapid reaction takes place.

$$14AgCNS + 7I_2 + 10H_2O \rightarrow 14\underline{AgI} + 12HCNS + H_2SO_4 + HCN + CO_2 + NH_4HSO_4$$

C. W. B. Normand and A. C. Cumming, J. Chem. Soc. (London), **101**, 1861 (1912) *57*

I_2

III-2497

AgF

Iodine reacts with silver fluoride to form iodine pentafluoride.

$$I_2 + 10AgF \rightarrow 2IF_5 + (10Ag)$$

McIvor, 26
Ref., R. Gerstl, Ber. [II], **8**, 1466 (1875)

I_2

III-2498

$AgNO_3$

An aqueous solution of I_2, treated with a solution of silver nitrate, yields a pale yellow liquid containing silver iodide in finely divided condition. When equivalent amounts of reactants are used about 96% of the material reacts as in equation (a). Some of the product reacts according to equation (b). Silver acetate reacts as the nitrate.

(a) $$AgNO_3 + I_2 + H_2O \rightarrow \underline{AgI} + HOI + HNO_3$$

(b) $$3HOI \rightarrow HIO_3 + 2HI$$

(c) $$CH_3COOAg + I_2 + H_2O \rightarrow AgI + HOI + CH_3COOH$$

H. S. Taylor, J. Chem. Soc. (London), **103**, 33, 35 (1913) *57*

I_2

III-2499

AgN_3

A triazide of iodine is obtained when a solution of iodine in benzene or ether reacts upon silver triazide.

$$2AgN_3 + 2I_2 \rightarrow 2IN_3 + (2\underline{AgI})$$

Hantzsch, Ber., **33**, 522 (1900)
Ref., Dennis and Browne, J. Am. Chem. Soc., **26**, 583 (1904) *1*

I_2

III-2500

Ag_2SO_3

Silver sulfite and iodine react in water to form iodide and sulfate.

$$H_2O + Ag_2SO_3 + I_2 \rightarrow 2\underline{AgI} + H_2SO_4$$

C. W. B. Normand and A. C. Cumming, J. Chem. Soc. (London), **101**, 1962 (1912) *57*

I_2

Al

Si **III-2501**

When a tube containing aluminum wire and iodine separated by an asbestos plug (the tube bent so that the middle portion is horizontal, and the ends point slightly downward) is evacuated, sealed, and heated so that the iodine at one end is at 180°, the metal in the middle is at 500°, and the other end is at 15°, aluminum iodide is formed. Silicon reacts similarly.

(a) $$2Al + 3I_2 \rightarrow 2AlI_3$$

(b) $$Si + 2I_2 \rightarrow SiI_4$$

M. Guichard, Compt. rend., **145**, 807 (1907)
Ref., E. Horton, J. Chem. Soc. (London), **94:2**, 31 (1908) *57*

I_2

$Ba(ClO_3)_2$ **III-2502**

When a neutral solution of barium chlorate is mixed with an equivalent amount of iodine in the presence of a small amount of HNO_3 or HCl at 85°C a rapid reaction occurs.

$$3I_2 + 3Ba(ClO_3)_2 + 3H_2O \rightarrow 3Ba(IO_3)_2 + HClO_3 + 5HCl$$

A. B. Lamb, W. C. Bray, and W. J. Geldard, J. Am. Chem. Soc., **42**, 1637 (1920) *16*

I_2

$Ba(ClO_3)_2$ **III-2503**

When slightly less than an equivalent amount of barium chlorate solution is mixed with iodine in the presence of a small amount of HCl or HNO_3 at 85°C the following reaction occurs.

$$6I_2 + 5Ba(ClO_3)_2 + 6H_2O \rightarrow 5Ba(IO_3)_2 + 2HIO_3 + 10HCl$$

A. B. Lamb, W. C. Bray and W. J. Geldard, J. Am. Chem. Soc., **42**, 1637 (1920) *16*

I_2

III-2504

CH_3COCH_3

Moisture promotes the acid-catalyzed iodination of acetone (b) by shifting the equilibrium in favor of iodoacetone.

(a) $CH_3COCH_3 \rightleftharpoons CH_3C(OH):CH_2$

(b) $CH_3C(OH):CH_2 + I_2 \rightleftharpoons CH_3CI(OH)CH_2I$

(c) $CH_3CI(OH)CH_2I \rightleftharpoons CH_3COCH_2I + HI$

M. P. Matuszak, Anal. Ed., **6**, 457 (1934) *44*

I_2

III-2505

CH_3COCH_3

Iodine atoms react with acetone to form iodoform. The iodine atoms are obtained through electrolysis, i.e., through anodic oxidation of iodine.

$$CH_3COCH_3 + 6I + H_2O \rightarrow CHI_3 + CH_3COOH + 3HI$$

K. Elbs and A. Herz, Z. Elektrochem., **4**, 114 (1897) *86*

I_2

III-2506

CH_3COSH

Iodine acts on thioacetic acid at ordinary conditions to form acetyl disulfide and hydriodic acid.

$$2CH_3COSH + I_2 \rightarrow (CH_3CO)_2S_2 + 2HI$$

N. Tarugi, Gazz. chim. ital., **27**[1], 158 (1897) *21*

I_2

III-2507

$C_2H_3O_2Ag$

When a solution of silver acetate is treated with iodine in equivalent amounts, silver iodide and silver iodate are formed. In excess iodine no iodate remains in the precipitate, but iodic acid is found in the filtrate.

(a) $6C_2H_3O_2Ag + 3H_2O + 3I_2 \rightarrow \underline{5AgI} + \underline{AgIO_3} + 6C_2H_3O_2H$

(b) $5C_2H_3O_2Ag + 3H_2O + 3I_2 \rightarrow \underline{5AgI} + HIO_3 + 5C_2H_3O_2H$

C. W. B. Normand, J. Chem. Soc. (London), **101**, 1853 (1912) *57*

I_2

C_2H_5OH **III-2508**

In the presence of alkali or carbonate and after heating, iodine reacts with alcohol to form iodoform. The same reaction can be brought about with aqueous KI-solution, where I_2 is formed anodically. If current density is small, the alcohol will be oxidized only to formic acid.

(a) $C_2H_5OH + 5I_2 + H_2O \rightarrow \underline{CHI_3} + CO_2 + 7HI$

(b) $C_2H_5OH + 4I_2 + H_2O \rightarrow \underline{CHI_3} + HCOOH + 5HI$

K. Elbs and A. Herz, Z. Elektrochem., **4**, 113, (1897) *86*

I_2

C_2H_5SH **III-2509**

Na_2SO_3

Sodium ethyl thiosulfate may be prepared by the action of iodine on a mixture of ethyl mercaptan and sodium sulfite.

$$I_2 + C_2H_5SH + Na_2SO_3 \rightarrow NaC_2H_5S_2O_3 + (NaI + HI)$$

D. F. Twiss, J. Chem. Soc. (London), **105**, 1692 (1914)
Ref., Spring and Legros, Ber., **15**, 1938 (1882) *57*

I_2

C_3H_3OH **III-2510**

Iodine reacts with propargyl alcohol in the presence of red phosphorus to form propargyl iodide.

$$6C_3H_3OH + 3I_2 + 2P \rightarrow 6C_3H_3I + (2H_3PO_3)$$

L. Henry, Ber., **8**:1, 398 (1875) *26*

I_2

$C_3H_4O_3$ **III-2511**

Pyruvic acid is oxidized quantitatively by iodine in strongly alkaline solution. The sodium hypoiodite formed acts both as an oxidizing and iodizing agent.

$$C_3H_4O_3 + 3I_2 + 5NaOH \rightarrow CHI_3 + 3NaI + Na_2C_2O_4 + 4H_2O$$

W. H. Hatcher and W. H. Mueller, Trans. Roy. Soc. (Canada) [III], **23**, 35 (1929) *29*

I_2 **III-2512**

NaOH

C_3H_6O

The oxidation of acetone by iodine in strongly alkaline solution is not quantitative, and therefore cannot be used for the estimation of acetone.

$$C_3H_6O + 3I_2 + 4NaOH \rightarrow CHI_3 + NaC_2H_3O_2 + 3NaI + 3H_2O$$

W. H. Hatcher and W. H. Mueller, Trans. Roy. Soc. (Canada) [III], **23**, 35 (1929) *69*

I_2 **III-2513**

C_6H_5OH

NaOH

Phenols in coke oven gas may be quantitatively determined by treatment with iodine and sodium hydroxide.

$$3I_2 + C_6H_5OH + 3NaOH \rightarrow C_6H_2I_3OH + 3NaI + 3H_2O$$

R. D. Scott, Anal. Ed., **3**, 67 (1931) *44*

I_2 **III-2514**

C_6H_5OH

Na_2CO_3

If sodium carbonate is added to a boiling solution of a phenol and iodine the following reaction takes place.

$$2C_6H_5OH + 6I_2 + 4Na_2CO_3 \rightarrow 2C_6H_2I_2O + 8NaI + 4CO_2 + 4H_2O$$

W. O. Emery, J. Ind. Eng. Chem., **13**, 538 (1921) *22*

I_2

C_6H_5ONa **III-2515**

When sodium phenolate reacts with iodine in presence of moisture, phenol and hypoiodous acid are first formed (a); the hypoiodous acid subsequently reacts with the phenol, giving the corresponding iodophenol (b).

(a) $C_6H_5ONa + I_2 + H_2O \rightarrow C_6H_5OH + NaI + IOH$

(b) $C_6H_5OH + IOH \rightarrow C_6H_4I \cdot OH + H_2O$

V. Cofman, J. Chem. Soc. (London), **115**:2, 1048 (1919) *48*

I_2

$C_7H_6O_3$ **III-2516**

KOH

If iodine and salicylic acid are mixed (both reactants being dissolved in alkali), iodosalicylic acid will be formed (a). The same compound is obtained by the action of iodine and iodic acid on salicylic acid (b).

(a) $C_7H_6O_3 + I_2 + KOH \rightarrow KI + H_2O + C_7H_5O_3I$

(b) $5C_7H_6O_3 + 2I_2 + HIO_3 \rightarrow 5C_7H_5O_3I + 3H_2O$

Kekulé, Ann., **131**, 221 (1864)
Ref., V. Cofman, J. Chem. Soc. (London), **115**:2, 1040 (1919) *48*

I_2

Cl^- **III-2517**

Br^-

Solubility determinations at 25° indicate equilibrium constants of approximate value 0.63 and 0.08 for the reversible reactions (*a*) and (*b*) shown below.

(a) $I_2 + Br^- \rightleftarrows I_2Br^-$

(b) $I_2 + Cl^- \rightleftarrows I_2Cl^-$

V. K. LaMer and M. H. Lewinsohn, J. Phys. Chem., **38**, 171 (1934) *7*

I_2

III-2518

Cl_2

Chlorine was passed over iodine in a tube to form iodine monochloride.

$$Cl_2 + I_2 \rightarrow 2ICl$$

W. E. Curtis and J. Patkowski, Trans. Roy. Soc. (London), **232A**, 396 (1934) *105*

I_2

III-2519

Cl_2

Iodine reacts violently with liquid chlorine, the trichloride being insoluble in liquid chlorine.

$$(I_2 + 3Cl_2 \rightarrow 2ICl_3)$$

K. H. Butler and D.McIntosh, Trans. Roy. Soc. (Canada) [III], **21**, 19 (1927) *69*

I_2

III-2520

Fe

When a tube containing iron wire and iodine separated by an asbestos plug, is evacuated, sealed, and heated so that the iron is kept at 500° and the iodine at 180°, ferrous iodide is formed and sublimes as red crystals. Uranium and nickel also form iodides under similar conditions.

(a) $$Fe + I_2 \rightarrow FeI_2$$

(b) $$2U + 3I_2 \rightarrow 2UI_3$$

(c) $$Ni + I_2 \rightarrow NiI_2$$

M. Guichard, Compt. rend., **145**, 807 (1907)
Ref., E. Horton, J. Chem. Soc. (London), **94:2**, 31 (1908) *57*

I_2

III-2521

HCOOH

Iodine and formic acid, in the presence of excess of potassium iodide, react to give hydrogen iodide and carbon dioxide.

$$HCOOH + I_2 \rightarrow 2HI + CO_2$$

D. L. Hammick and M. Zvegintzov, J. Chem. Soc. (London), **2**, 1105 (1926) *65*

I_2

HCOONa **III-2522**

Sodium formate is easily oxidized by iodine at ordinary temperature forming sodium iodide, hydriodic acid and carbon dioxide.

$$HCOONa + I_2 \rightarrow NaI + HI + CO_2$$

N. R. Dhar, J. Chem. Soc. (London), **111**, 726 (1917) *19*

I_2

HCl_3 **III-2523**

In the "iodate" methods of volumetric analysis there is usually added to a hydrochloric acid solution of the reducing substance to be analyzed a standard solution of an iodate until the iodine at first liberated is oxidized to iodine monochloride. The end-point is determined by the disappearance of the iodine color in an immiscible organic solvent, usually carbon tetrachloride or chloroform.

$$I_2 + 2HCl + O(\text{from iodate}) \rightarrow 2ICl + H_2O$$

E. H. Swift, J. Am. Chem. Soc., **52**, 894 (1930) *16*

I_2

HCl **III-2524**

KIO

Iodine is oxidized to iodine monochloride by a hydrochloric acid solution of an iodate.

$$2I_2 + 6HCl + KIO_3 \rightarrow 5ICl + 3H_2O + KCl$$

E. H. Swift, J. Am. Chem. Soc., **52**, 900 (1930)
Ref., Schoonover and Furmen, J. Am. Chem. Soc., **55**, 3124 (1933) *1*

I_2

$HClO_3$ **III-2525**

When iodine is added to chloric acid, chlorine is liberated.

$$2HClO_3 + I_2 \rightarrow 2HIO_3 + Cl_2$$

M. Gomberg, J. Am. Chem. Soc., **45**, 407 (1923) *19*

I_2 **III-2526**

$HClO_3$

To obtain iodic acid, iodine was oxidized by a small excess (3%) of 24–26% chloric acid solution.

$$I_2 + 2HClO_3 \rightarrow 2HIO_3 + Cl_2$$

A. B. Lamb, W. C. Bray, and W. J. Geldard, J. Am. Chem. Soc., **42**, 1647 (1920) *1*

I_2 **III-2527**

$HClO_4$

When iodine is added to perchloric acid the iodine replaces the chlorine.

$$2HClO_4 + I_2 \rightarrow (2HIO_4 + Cl_2) \rightarrow 2HIO_3 + O_2 + Cl_2$$

M. Gomberg, J. Am. Chem. Soc., **45**, 407 (1923) *16*

I_2 **III-2528**

HI

Iodine or metallic iodide dissolved in hydriodic acid yields a periodide.

$$(I_2 + HI \rightarrow HI_3)$$

Johnson, Trans. J. Chem. Soc. (London), **31**, 249 (1877)
Dawson, Trans. J. Chem. Soc. (London), **81**, 524 (1902) and **85**, 796, (1904)
Ref., Tinkler, J. Chem. Soc. (London), **91**, 996 (1907) *109*

I_2 **III-2529**

HIO_3

CH_3CHO

In presence of iodic acid, iodine reacts with acetaldehyde and its homologues to form mono-substituted derivatives.

$$5CH_3CHO + 2I_2 + HIO_3 \rightarrow 5CH_2ICHO + 3H_2O$$

H. M. Dawson, J. Chem. Soc. (London), **105**, 1276 (1914)
Ref., Chautard, Ann. chim. phys., **16** [iv], 145 (1889) *57*

I_2

HIO_3 **III-2530**

HCl

Iodine trichloride is formed when a mixture containing finely ground iodine and iodic acid is slowly added to twice the theoretical volume of hydrochloric acid and shaken.

$$3HIO_3 + I_2 + 15HCl \rightarrow 5ICl_3 + 9H_2O$$

L. B. Howell and W. A. Noyes, J. Am. Chem. Soc., **42**, 1009 (1920) *1*
16

I_2

HIO_3 **III-2531**

HCl

In the volumetric determination of vanadium as vanadate, the vanadium is reduced by iodide in 6 molal HCl solution and the liberated iodine converted into iodine monochloride by titration with iodate.

$$2I_2 + HIO_3 + 5HCl \rightarrow 5ICl + 3H_2O$$

E. H. Swift and R. H. Hoeppel, J. Am. Chem. Soc., **51**, 1366 (1929) *16*

I_2

HNO_3 **III-2532**

Nitric acid oxidizes iodine to the dioxide.

$$8HNO_3 + I_2 \rightarrow I_2O_4 + (4H_2O + 8NO_2)$$

Millon, Ann. chim. phys., **12**, 333
Ref., M. M. P. Muir, J. Chem. Soc. (London), **95**, 656 (1909) *57*

I_2

HNO_3 **III-2533**

Fuming nitric acid reacts with iodine to form iodic acid, but the yields are very unsatisfactory.

$$I_2 + 4HNO_3 \rightarrow 2HIO_3 + (3NO + NO_2 + H_2O)$$

M. Guichard, Compt. rend., **148**, 924 (1909) *38*

I_2 **III-2534**

$HO \cdot C_6H_4 \cdot COOAg$

If iodine reacts with silver salicylate, the first step of the reaction will consist in the formation of salicylic acid, silver iodide and hypoiodous acid, (a). Subsequently, the hypoiodous acid formed reacts with salicylic acid to form the corresponding iodo-salicylic acid, (b).

(a) $OH \cdot C_6H_4 \cdot COOAg + I_2 + H_2O \rightarrow OH \cdot C_6H_4 \cdot COOH + \underline{AgI} + HIO$

(b) $OH \cdot C_6H_4 \cdot COOH + IOH \rightarrow OH \cdot C_6H_3I \cdot COOH + H_2O$

V. Cofman, J. Chem. Soc. (London), **115**:2, 1048 (1919) *48*

I_2 **III-2535**

$HO \cdot C_6H_4 \cdot COOAg$

When iodine reacts with silver salicylate, iodosalicylic acid is formed and silver iodide precipitates.

$$OH \cdot C_6H_4 \cdot COOAg + I_2 \rightarrow OH \cdot C_6H_3I \cdot COOH + \underline{AgI}$$

Birnbaum, Ber., **15**, 459 (1882)
Schall, Ber., **16**, 1897 (1883)
Ref., V. Cofman, J. Chem. Soc. (London), **115**:2, 1041 (1919) *48*

I_2 **III-2536**

$HPO_4^{=}$

Iodine reacts with hydrogen phosphate ion, forming iodide ion, iodate ion and di-hydrogen-phosphate ion.

$$3I_2 + 6HPO_4^{=} + 3H_2O \rightarrow 5I^- + IO_3^- + 6H_2PO_4^-$$

Skrabal and Gruber, Monatsh., **37**, 543 (1916)
Ref., V. Cofman, J. Chem. Soc. (London), **115**:2, 1047 (1919) *48*

I_2 **III-2537**

H_2O

Iodine is hydrolyzed in aqueous solution producing hydriodic acid, hypoiodous acid, and iodic acid.

(a) $I_2 + H_2O \rightarrow HI + HIO$

(b) $3I_2 + 3H_2O \rightarrow 5HI + HIO_3$

Bray, J. Am. Chem. Soc., **32**, 932 (1910)
Ref., C. W. B. Normand, J. Chem. Soc. (London), **101**, 1853 (1912) *57*

I_2

H_2O **III-2538**

Iodine reacts with water when heated in sunlight.

$$2I_2 + 2H_2O \rightarrow 4HI + O_2$$

G. Lemoine, Compt. rend., **85**, 144 (1877) *25*

I_2

H_2O **III-2539**

Iodine dissolved in alcohol reacts with water to form HI and HOI especially in the presence of unsaturated organic compounds.

$$H_2O + I_2 \rightarrow HI + HOI$$

E. Galle and R. Klatt, Brenn. Chem., **14**, 321 (1933) *11*

I_2

H_2O **III-2540**

HNO_3

Iodine is oxidized by nitric acid.

$$I_2 + 6HNO_3 + H_2O \rightarrow 2HIO_4 + 6HNO_2$$

M. Schlötter, Z. anorg. Chem., **45**, 270 (1905) *25*

I_2

H_2O **III-2541**

SO_2

CH_3OH

With water, a methanol solution of Karl Fischer reagent produces hydriodic and methyl sulfuric acids.

$$I_2 + H_2O + SO_2 + CH_3OH \rightarrow 2HI + HSO_4CH_3$$

J. Mitchell, Jr., D. M. Smith, E. C. Ashby, and W. M. D. Bryant, J. Am. Chem. Soc., **63**, 2927 (1941) *16*

I_2 **III-2542**

H_2O_2

Hydrogen peroxide oxidizes iodine to iodic acid.

$$5H_2O_2 + I_2 \rightarrow 2HIO_3 + 4H_2O$$

W. C. Bray, J. Am. Chem. Soc., **43**, 1262 (1921) *1*

I_2 **III-2543**

H_2S

Iodine is dissolved in carbon disulfide and water is placed on top, the hydrogen sulfide is passed to the bottom of the vessel into the iodine solution.

$$(H_2S + I_2 \rightarrow 2HI + S)$$

C. Winkler, Chem. News, **2**, 144 (1868) *101*

I_2 **III-2544**

H_2S

When hydrogen sulfide is passed through a solution of iodine, hydrogen iodide and sulfur are formed.

$$H_2S + I_2 \rightarrow 2HI + S$$

P. Poetschke, J. Ind. Eng. Chem., **5**, 981 (1913) *22*

I_2 **III-2545**

H_2S

If an aqueous suspension of iodine be saturated with hydrogen sulfide and the resulting solution be distilled, hydriodic acid (d. 1.7) will be collected in the fraction boiling between 123° and 127°.

$$I_2 + H_2S \rightarrow 2HI + S$$

J. T. Hewitt and W. J. Jones, J. Chem. Soc. (London), **115:1**, 193 (1919) *48*

I_2

H_2S_3 **III-2546**

When a solution of iodine in hydrogen persulfide is heated in a sealed tube, liquid hydrogen iodide is formed.

$$I_2 + H_2S_3 \rightarrow 2HI + \underline{(3S)}$$

W. Ramsay, J. Chem. Soc. (London), **27**, 857 (1874) *48*

I_2

H_2S_3 **III-2547**

When a solution of iodine in hydrogen persulfide is allowed to stand, iodine sulfide is formed with elimination of hydrogen sulfide.

$$I_2 + 2H_2S_3 \rightarrow 2IS_2 + 2H_2S$$

W. Ramsay, J. Chem. Soc. (London), **27**, 360 (1874) *48*

I_2

H_2SO_3 **III-2548**

Iodine oxidizes sulfurous acid solution.

$$H_2SO_3 + I_2 + H_2O \rightarrow 2HI + H_2SO_4$$

J. B. Ferguson, J. Am. Chem. Soc., **39**, 365 (1917) *1*

I_2

H_3PO_2 **III-2549**

The reaction between iodine and hypophosphorous acid (in mineral acid solution) is as in (a). In sodium carbonate solution the reaction is as in (b).

(a) $$H_3PO_2 + I_2 + H_2O \rightarrow H_3PO_3 + 2HI$$

(b) $$H_3PO_3 + I_2 + H_2O \rightarrow H_3PO_4 + 2HI$$

Rupp and Finck, Archiv. Pharm., **240**, 663 (1902) *109*
Ref., Steele, J. Chem. Soc. (London), **91**, 1641, (1907) *108*

I_2

III-2550

H_3PO_3

$NaHCO_3$

If iodine is reacted with phosphorous acid in the presence of sodium hydrogen carbonate the resulting compounds are phosphoric acid and hydriodic acid.

$$H_3PO_3 + I_2 + H_2O \rightarrow H_3PO_4 + 2HI$$

B. D. Steele, J. Chem. Soc. (London), **91**:2, (1641) (1907) *108*

I_2

III-2551

$Hg(CN)_2$

Cyanogen iodide is produced when resublimed iodine dissolved in ether is treated with an excess of mercuric cyanide and shaken vigorously.

$$Hg(CN)_2 + 2I_2 \rightarrow 2ICN + HgI_2$$

Badger and Xho-Chow Woo, J. Am. Chem. Soc. **53**, 2573 (1931) *1*

I_2

III-2552

$Hg(ClO_4)_2$

If mercuric perchlorate is added to a solution of iodine autoxidation of the iodine occurs.

$$6I_2 + 6Hg(ClO_4)_2 + 6H_2O \rightarrow 5HgI_2 + Hg(IO_3)_2 + 12HClO_4$$

N. H. Furman and C. O. Miller, J. Am. Chem. Soc., **59**, 153 (1937) *16*

I_2

III-2553

HgO

With mercuric oxide, iodine undergoes autoxidation.

$$6I_2 + 6HgO \rightarrow 5HgI_2 + Hg(IO_3)_2$$

N. H. Furman and C. O. Miller, J. Am. Chem. Soc., **59**, 153 (1937) *16*

I_2

KCN **III-2554**

Iodine cyanide is prepared by adding iodine to a moderately concentrated solution of potassium cyanide and extracting the resulting mixture with ether. The iodine color has disappeared.

$$2KCN + 2I_2 \rightarrow 2ICN + 2KI$$

D. M. Yost and W. E. Stone, J. Am. Chem. Soc., **55**, 1889 (1933) *1*

I_2

KI **III-2555**

Potassium iodide and iodine in nitrobenzene solution react to form polyhalides, the molecular weight of which depends upon the relative amounts of material present.

(a) $KI + I_2 \rightarrow KI_3$

(b) $KI + 2I_2 \rightarrow KI_5$

(c) $KI + 3I_2 \rightarrow KI_7$

(d) $KI + 4I_2 \rightarrow KI_9$

H. M. Dawson and C. G. Jackson, J. Chem. Soc. (London), **93**, 2063 (1908) *57*

I_2

KI **III-2556**

Solid triiodides of rubidium, cesium and ammonium have been reported, and their existence remains unquestioned. Solid KI_3 is not stable at 25°C although it exists in reversible equilibrium in potassium iodide solutions. There is also evidence that relatively small amounts of a still more complex polyiodide, possibly I_{14}^-, is formed in concentrated solutions of KI saturated with iodine. CsI_4 is also formed under similar circumstances.

(a) $I^- + I_2 \rightleftarrows I_3^-$

(b) $2I^- + 6I_2^- \rightleftarrows I_{14}^-$

G. Jones, J. Phys. Chem., **34**, 690 (1930)
T. R. Briggs, J. Phys. Chem., **34**, 2259, 2266, (1930)
N. Rae, J. Phys. Chem., **35**, 1802 (1931)
N. Bancroft, G. Sherer and L. Gould, J. Phys. Chem., **35**, 784 (1931) *85*

I_2

III-2557

KI

The temperature-composition diagrams of the I_2-KI-H_2O system have been determined in detail. The stable, solid phases (besides original components) are: $KI_3 \cdot H_2O$, $KI_3 \cdot 2H_2O$ (two polymorphic modifications), and $KI_7 \cdot H_2O$. The first and last are stable at 25°C.

(a) $$KI + I_2 + H_2O \rightarrow KI_3 \cdot H_2O$$

(b) $$KI + I_2 + 2H_2O \rightarrow KI_3 \cdot 2H_2O$$

(c) $$KI + 3I_2 + H_2O \rightarrow KI_7 \cdot H_2O$$

Briggs, Clack, Ballard and Sassaman, J. Phys. Chem., **44**, 372 (1940)
H. W. Foote and W. Bradley, J. Phys. Chem., **36**, 673, 1713 (1932) *85*

I_2

III-2558

KI

HCOONa

Iodine dissolved in potassium iodide solution oxidizes formates. A salt of a weak acid is added to reduce the concentration of hydrogen ion produced. Excess iodide ion retards the reaction.

(a) $$KI + I_2 \rightarrow KI_3$$

(b) $$KI_3 + HCOONa \rightarrow NaI + HI + CO_2 + KI$$

N. Dhar, J. Chem. Soc. (London), **111**, 726 (1917) *57*

I_2

III-2559

KOH

If alkali is added to an aqueous solution of iodine, potassium iodide, potassium hypoiodite and water are the products of the reaction.

$$2KOH + I_2 \rightarrow KI + KOI + H_2O$$

R. L. Taylor, J. Chem. Soc. (London), **77:2**, 726 (1900) *103*

I_2

KOH **III-2560**

Iodine reacts with potassium hydroxide under standard conditions to give potassium iodate as follows:

(a) $$I_2 + 2KOH \rightarrow 2KIO + H_2$$

(b) $$3KIO \rightarrow 2KI + KIO_3$$

G. Gallo, Gazz. chim. ital., **36**[2], 118 (1906) *21*

I_2

KOH **III-2561**

$CH_3C(H)(OH)SO_3K$

If the pH is closely watched, iodine will react with potassium hydroxide and the addition product of potassium bisulfite and acetaldehyde, under quantitative formation of potassium iodide and iodoform.

$$4I_2 + 7KOH + CH_3C(H)(OH)SO_3K \rightarrow KHSO_4 + HCOOK + \underline{HCI_3} + 5H_2O + 5KI$$

W. Müller-Parcham, Z. physiol. Chem., **218**, 209 (1933) *14*

I_2

$KSCSN_3$ **III-2562**

Azidocarbondisulfide precipitates when iodine reacts with potassium azidodithiocarbonate.

$$2KSCSN_3 + I_2 \rightarrow \underline{(SCSN_3)_2} + 2KI$$

A. W. Browne and A. B. Hoel, J. Am. Chem. Soc., **44**, 2110 (1922) *1*

I_2

$K_2C_2O_4$ **III-2563**

Oxalates are oxidized by iodine. The velocity of reaction is greatly increased by sunlight.

$$K_2C_2O_4 + I_2 \rightarrow 2KI + 2CO_2$$

N. Dhar, J. Chem. Soc. (London), **111**, 748 (1917) *57*

I_2

III-2564

NH_2OH

Iodine oxidizes hydroxylamine in neutral solution in the presence of disodium phosphate.

$$2NH_2OH + 2I_2 \rightarrow N_2O + 4HI + H_2O$$

W. Meyeringh, Ber., **10**, 1940 (1877)
T. Haga, J. Chem. Soc. (London), **51**, 794 (1887) *1*
Bray, Simpson and MacKenzie, J. Am. Chem. Soc., **41**, 1363 (1919) *86*
B. Suler, Z. Elektrochem., **7**, 831 (1901) *102*
H. O. Jones and F. W. Carpenter, J. Chem. Soc. (London), **83**, 1395 (1903)

I_2

III-2565

NH_2OH

H_2O

The well known reaction between iodine and hydroxylamine is quantitative if sulfanilic acid is present to catch immediately the nitrous acid formed.

$$2I_2 + H_2O + NH_2OH \rightarrow HNO_2 + 4HI$$

G. Endres and L. Kaufmann, Z. physiol. Chem., **243**, 144, (1936) *14*

I_2

III-2566

NH_3

Iodine is very soluble in a small volume of liquid ammonia and the solution contains iodine-amide and ammonium iodide.

$$I_2 + 2NH_3 \rightleftharpoons INH_2 + NH_4I$$

F. W. Bergstrom, J. Am. Chem. Soc., **48**, 2320 (1926) *16*

I_2

NH_4I **III-2567**

A single polyiodide, NH_4I_3, is stable in the NH_4-I_2 system. This salt melts incongruently at 175°C. In the presence of water a tri-hydrate of this tri-iodide and a pentaiodide, $NH_4I_5 \cdot H_2O$, are also found. Both melt incongruently, the former at 7.0°C and the latter at 9.8°C.

(a) $$NH_4I + I_2 \rightarrow NH_4I_3$$

(b) $$NH_4I + I_2 + 3H_2O \rightarrow NH_4I_3 \cdot 3H_2O$$

(c) $$NH_4I + 2I_2 + H_2O \rightarrow NH_4I_5 \cdot H_2O$$

Briggs, Ballard, Alrich and Wikswo, J. Phys. Chem., **44**, 349 (1940) *85*

I_2

NH_4OH **III-2568**

HCl

Iodine monochloride is prepared by the action of concentrated hydrochloric acid upon nitrogen tri-iodide formed by digesting iodine in ammonium hydroxide.

(a) $$6I_2 + 4NH_4OH \rightarrow 2NH_3 \cdot NI_3 + 4H_2O + 6HI$$

(b) $$NH_3 \cdot NI_3 + 5HCl \rightarrow 3ICl + 2NH_4Cl$$

L. B. Howell and W. A. Noyes, J. Am. Chem. Soc., **42**, 1009 (1920) *1*

I_2

N_2H_4 **III-2569**

Hydrazine changes iodine to hydriodic acid and sets nitrogen free.

$$2I_2 + N_2H_4 \rightarrow 4HI + N_2$$

G. Holst, Sv. Kem. Tidsk., **43**, 16 (1931) *10*

I_2

N_2O_5 **III-2570**

Iodine, moistened with fuming nitric acid, reacts with nitric anhydride (from fuming nitric acid and phosphorus pentoxide) to form iodic anhydride in 40% yield.

$$I_2 + N_2O_5 \rightarrow I_2O_5 + (N_2)$$

M. Guichard, Compt. rend., **148**, 925 (1909) *38*

I_2

III-2571

Na

If iodine is treated with sodium in liquid ammonia, iodide ions are formed.

$$I_2 + 2Na^+ + e^- \rightarrow 2Na^+ + 2I^-$$

C. A. Kraus, J. Franklin Inst., **212**, 559 (1931) *16*

I_2

III-2572

$NaClO_3$

Cl_2

KOH

Trisodium paraperiodate is formed when iodine is oxidized to sodium iodate and this salt oxidized to periodate by chlorine in an alkaline solution.

(a) $$I_2 + 2NaClO_3 \rightarrow 2NaIO_3 + Cl_2$$

(b) $$6NaIO_3 + 3Cl_2 + 6KOH \rightarrow 2Na_3H_2IO_6 + 4KClO_3 + 2HI + 2KCl + I_2$$

H. H. Willard and J. J. Thompson, J. Am. Chem. Soc., **56**, 1828 (1934) *1*

I_2

III-2573

$NaHCO_3$

H_2O_2

Iodine, hydrogen peroxide, and sodium bicarbonate react to form sodium iodide, carbon dioxide, and oxygen.

$$I_2 + 2NaHCO_3 + H_2O_2 \rightarrow 2NaI + 2CO_2 + 2H_2O + O_2$$

A. V. Harcourt and W. Esson, Trans. Roy. Soc. (London), **186**, 855 (1895) *105*

I_2

$NaHSO_3$ **III-2574**

Na_2CO_3

Iodine treated with a solution of sodium hydrogen sulfite and sodium carbonate is changed to sodium iodide, the other products being sodium sulfate, carbon dioxide and water.

$$2NaHSO_3 + 3Na_2CO_3 + 2I_2 \rightarrow 4NaI + 2Na_2SO_4 + 3CO_2 + H_2O$$

A. G. Ekstrand, Sv. Kem. Tidsk., **5**, 93 (1893) *10*

I_2

NaI **III-2575**

Three different solid polyiodides have been found in the system $NaI \cdot I_2 \cdot H_2O$. No solid tri-iodide of sodium has been found at any temperature, although tri-iodides of NH_4, K, Rb, and Cs are readily obtained under similar conditions.

(a) $4NaI + 5I_2 + 13\text{–}15H_2O \rightarrow Na_4I_{14} \cdot 13\text{–}15H_2O$ below 16.4°C

(b) $5NaI + 3I_2 + 17\text{–}19H_2O \rightarrow Na_5I_{11} \cdot 17\text{–}19H_2O$ below 2°C

(c) $2NaI + 3I_2 + 10\text{–}11H_2O \rightarrow Na_2I_8 \cdot 10\text{–}11H_2O$ below −7°C

T. R. Briggs, W. F. Geigle, and J. L. Eaton, J. Phys. Chem., **45**, 612 (1941) *85*

I_2

(NaOH) **III-2576**

Iodine reacts with alkali to yield hypoiodous acid and iodide. With excess alkali the hypoiodide is formed.

(a) $I_2 + (NaOH) \rightarrow HOI + (NaI)$

(b) $HOI + (NaOH) \rightarrow (NaOI) + H_2O$

H. S. Taylor, J. Chem. Soc. (London), **103**, 32 (1913) *57*

I_2

NaOH **III-2775**

The action of iodine on alkali is connected with the equilibria of the following two reactions:

(a) $I_2 + OH^- \rightleftharpoons HOI + I^-$

(b) $HOI + OH^- \rightleftharpoons OI^- + H_2O$

F. Foerster, Z. Elektrochem., **9**, 216 (1903) *86*

I_2

III-2578

NaOH

Iodine interacts with alkali in solution to form iodate and iodide.

$$3I_2 + 6NaOH \rightarrow NaIO_3 + 5NaI + 3H_2O$$

G. M. Kline and S. F. Acree, Anal. Ed., **2**, 413 (1930) *33*

I_2

III-2579

NaOH

Free iodine in a solid soap will disappear in aqueous solution, owing to liberation of caustic soda by hydrolysis, and it can be shown that the solution of a soap claimed to contain iodine has no action on the starch reagent.

$$6NaOH + 3I_2 \rightarrow 5NaI + NaIO_3 + 3H_2O$$

J. Glenn, Jr., Perfumery and Cosmetics, p. 529 Aug. 1936
Ref., Abstracts, Oil and Soap, **13**, 11, 295 (1936) *111*

I_2

III-2580

NaOH

Hypoiodite and iodides are formed by the reaction between sodium hydroxide and iodine.

$$2NaOH + I_2 \rightarrow NaOI + NaI + H_2O$$

G. Heinemann and H. W. Rahn, Anal. Ed., **9**, 458 (1937) *44*

I_2

III-2581

NaOH

Cl_2

A white precipitate which is converted into silver periodate is produced when a rapid stream of chlorine is passed through a solution of sodium hydroxide and iodine.

(a) $12NaOH + 7Cl_2 + I_2 \rightarrow \underline{2H_3Na_2IO_6} + (6HCl + 8NaCl)$

(b) $H_3Na_2IO_6 + 3AgNO_3 \rightarrow \underline{Ag_3IO_5} + (HNO_3 + 2NaNO_3 + H_2O)$

H. L. Wells, Am. Chem. J., **25**, 279 (1901) *1*

I_2

NaOH **III-2582**

RCHO

When alkali is added to a mixture of iodine and aldose sugar, the following reactions are initiated.

(a) $$2NaOH + I_2 \rightarrow NaIO + NaI + H_2O$$

(b) $$NaIO + RCHO + NaOH \rightarrow RCOONa + NaI + H_2O$$

(c) $$3NaIO \rightarrow NaIO_3 + 2NaI$$

R. E. Lothrup and R. L. Holmes, Anal. Ed., **3**, 334 (1931) *44*

I_2

Na_2HPO_4 **III-2583**

Iodine dissolves in a solution of disodium hydrogen phosphate with formation of sodium-dihydrogen phosphate, sodium iodide and hypoiodous acid (a). The latter decomposes further into iodic acid, iodine and water, (b).

(a) $$Na_2HPO_4 + I_2 + H_2O \rightarrow NaH_2PO_4 + NaI + HIO$$

(b) $$5HIO \rightarrow HIO_3 + 2I_2 + 2H_2O$$

V. Cofman, J. Chem. Soc. (London), **115**:**2**, 1047 (1919) *48*

I_2

Na_2O_2 **III-2584**

Sodium peroxide mixed with iodine and then steadily cooled will produce sodium iodate and sodium iodide.

$$3Na_2O_2 + 3I_2 \rightarrow 2NaIO_3 + 4NaI$$

A. Longi and L. Banovia, Gazz. chim. ital., **28**[1], 334 (1898) *21*

I_2

Na_2S **III-2585**

$Na_2S_2O_3$

A difference exists between the stoichiometric iodine demands of sulfide and thiosulfate sulfur.

(a) $$Na_2S + I_2 \rightarrow 2NaI + S$$

(b) $$2Na_2S_2O_3 + I_2 \rightarrow Na_2S_4O_6 + 2NaI$$

G. Heinemann and H. W. Rahn, Anal. Ed., **9**, 458 (1937) *44*

I_2 **III-2586**

$Na_2S_2O_3$

Iodine in solution was analyzed for by sodium thiosulfate, forming sodium iodide and sodium tetrathionate.

$$2Na_2S_2O_3 + I_2 \rightarrow 2NaI + Na_2S_4O_6$$

L. Mond, W. Ramsay, and J. Shields, Trans. Roy. Soc. (London), **186**, 818 (1895) *105*

I_2 **III-2587**

$Na_2S_2O_3$

Free iodine may be titrated with a solution of sodium thiosulfate.

$$3I_2 + 6Na_2S_2O_3 \rightarrow 6NaI + 3Na_2S_4O_6$$

M. Blanchetiere, Bull. soc. chim. [4], **19**, 304 (1916) *31*

I_2 **III-2588**

OH^-

The first reaction which takes place when iodine is dissolved in alkali results in the formation of hypoiodous acid and iodide ion. Equilibrium is attained rapidly, and if the alkali is concentrated, the concentration of free iodine is negligible. The subsequent decomposition of the hypoiodous acid is a relatively slow reaction.

$$I_2 + OH^- \rightarrow IOH + I^-$$

V. Cofman, J. Chem. Soc., (London), **115:2**, 1042 (1919) *48*

I_2 **III-2589**

O_2

Iodine is heated in a tube with air or oxygen to a high temperature.

$$4I + 5O_2 \rightarrow 2I_2O_5$$

Sementini, Ann., **4**, 170 (1832) *20*

I_2

RCHO **III-2590**

The determination of aldose sugars by titration with iodine-alkali reagents is based on the following reactions.

(a) $RCHO + I_2 + 3NaOH \rightarrow RCOONa + 2NaI + 2H_2O$

(b) $6I + 6NaOH \rightarrow NaIO_3 + 5NaI + 3H_2O$

G. M. Kline and S. F. Acree, Anal. Ed., **2**, 413 (1930) *44*

I_2

RCHO **III-2591**

NaOH

Aldehydes are quantitatively oxidized to acids by iodine.

$$I_2 + 3NaOH + RCHO \rightarrow RCOONa + 2NaI + 2H_2O$$

R. E. Lothrup and R. L. Holmes, Anal. Ed., **3**, 334 (1931) *44*

I_2

RbI **III-2592**

The stable solid phases in the system RbI-I_2, between 60°C and the boiling point of the saturated melt (238°C), are iodine, rubidium iodate and rubidium tri-iodide. The per-iodide melts incongruently at 188°C.

$$RbI + I_2 \rightarrow RbI_3$$

T. R. Briggs and E. S. Patterson, J. Phys. Chem., **36**, 2624 (1932) *85*

I_2

RbI **III-2593**

At 25°C and 1 atmosphere there are only three true polyiodides of the alkali metals, RbI_3, CsI_3, and CsI_4. Other polyiodides reported are solvated compounds. Various ternary polyiodides with benzene and toluene have been reported.

(a) $RbI + I_2 \rightarrow RbI_3$

(b) $CsI + I_2 \rightarrow CsI_3$

(c) $KI + I_2 + H_2O \rightarrow KI_3 \cdot H_2O$

(d) $KI + 4I_2 + 3C_6H_6 \rightarrow KI_9 \cdot 3C_6H_6$

N. S. Grace, J. Phys. Chem., **37**, 359 (1933) *85*

I_2 **III-2594**

SO_2

A methyl alcohol solution of iodine, sulfur dioxide and pyridine react with water to form acids.

$$2H_2O + SO_2 + I_2 \rightarrow H_2SO_4 + 2HI$$

H. Levin, K. Uhrig, and F. M. Roberts, Anal. Ed., **17**, 212 (1945) *44*

I_2 **III-2595**

SO_2

In a determination of water in fats, 254 g. I_2 in 5l dry MeOH and 790 g. pyridine are prepared; after cooling, 192 g. dry SO_2 are added. A sample of 20–50 g. of oil is dissolved in decalin and MeOH mixt., and treated with the iodine soln. The reaction proceeds until the water in the fat is used up. The iodine used is determined by difference.

$$I_2 + SO_2 + 2H_2O \rightarrow H_2SO_4 + 2HI$$

H. P. Kauffmann and S. Funke, Fette und Seifen, **44**, 345–6 (1937) *111*
Ref., Oil and Soap, **14**, 12, 328 (1937)

I_2 **III-2596**

SO_2

H_2O

Iodine is transformed to hydriodic acid by reacting it with sulfur dioxide and water. The reaction is reversible.

$$SO_2 + I_2 + H_2O \leftrightarrows SO_3 + 2HI$$

Chem. News, **9**, 99 (1864) *101*

I_2 **III-2597**

TlI

Thallium iodide forms two higher iodides at 6°C in the presence of benzene or toluene as solvents.

$$TlI + I_2 \rightarrow TlI_3$$
$$6TlI + I_2 \rightarrow Tl_6I_8$$

H. W. Foote, and M. Fleischer, J. Phys. Chem., **44**, 651 (1940) *85*

I_2

Aldoses **III-2598**

NaOH

In alkaline solution iodine is reduced to iodide by aldose sugars.

$$RCHO + I_2 + 3NaOH \rightarrow RCOONa + 2NaI + 2H_2O$$

G. M. Kline and S. F. Acree, Anal. Ed., **2**, 413 (1930) *33*

I_2

Xylose **III-2599**

In alkaline solution iodine interacts with xylose forming iodide and a salt of xylonic acid. This reaction is general for aldoses; 14 references on the reaction given.

$$C_4H_9O_4CHO + 3NaOH + I_2 \rightarrow C_4H_9O_4COONa + 2NaI + 2H_2O$$

C. S. Slater and S. F. Acree, Anal. Ed., **2**, 274 (1930) *33*

I_2

Δ **III-2600**

At temperatures above 950°C, iodine is dissociated into monatomic iodine.

$$I_2 \rightarrow 2I$$

W. C. M. Lewis, J. Chem. Soc. (London), **111**, 1091 (1917) *57*

I_2O_4

$H_2C_2O_4$ **III-2601**

Iodine dioxide reacts with an aqueous solution of oxalic acid in the presence of sulfuric acid at 100°.

$$I_2O_4 + 4H_2C_2O_4 \rightarrow 4H_2O + 8CO_2 + I_2$$

F. Fichter and F. Rohner, Ber., **42**, 4093 (1909) *25*

I_2O_4 **III-2602**

$H_2C_2O_4$

Oxalic acid reduces iodine dioxide to iodine.

$$I_2O_4 + 4H_2C_2O_4 \rightarrow 4H_2O + 8CO_2 + I_2$$

M. M. P. Muir, J. Chem. Soc. (London), **95**, 656 (1909) *1*
Ref., J. L. Howe, J. Am. Chem. Soc., **31**, 1293 (1909) *108*

I_2O_4 **III-2603**

H_2O

Diiodine tetroxide reacts slowly with cold water and quickly with hot water to form iodic acid and free iodine.

$$5I_2O_4 + 4H_2O \rightarrow 8HIO_3 + I_2$$

M. M. P. Muir, J. Chem. Soc. (London), **95**:1 657 (1909) *103*
Ref., J. L. Howe, J. Am. Chem. Soc., **31**, 1293 (1909) *57*
1

I_2O_4 **III-2604**

KOH

Iodine dioxide reacts with an aqueous solution of potassium hydroxide.

$$3I_2O_4 + 6KOH \rightarrow 5KIO_3 + KI + 3H_2O$$

H. Kappeler, Ber., **44**, 3496 (1911) *25*

I_2O_4 **III-2605**

KOH

Diiodine tetroxide reacts with aqueous potassium hydroxide to form potassium iodate, potassium iodide, and water.

$$3I_2O_4 + 6KOH \rightarrow 5KIO_3 + KI + 3H_2O$$

M. M. P. Muir, J. Chem. Soc. (London), **95**:1 659 (1909) *103*

I_2O_4

SO_3 **III-2606**

Iodine dioxide is placed in a tube to which sulfur trioxide is added, and the tube is sealed and heated at 80° for eight hours. A pale yellow solid addition compound is formed.

$$I_2O_4 + 3SO_3 \rightarrow I_2O_4 \cdot 3SO_3$$

M. M. P. Muir, J. Chem. Soc. (London), **95**, 659 (1909) *57*

$I_2O_4 \cdot 3SO_3$

Δ **III-2607**

The iodine dioxide-sulfur trioxide addition compound decomposes when heated to 120°, (a). It also decomposes when exposed to the air for a day or two, (b).

(a) $$I_2O_4 \cdot 3SO_3 \rightarrow I_2 + 2O_2 + 3SO_3$$

(b) $$4I_2O_4 \cdot 3SO_3 + 14H_2O \rightarrow 2I_2 + 4HIO_3 + (12H_2SO_4 + 3O_2)$$

M. M. P. Muir, J. Chem. Soc. (London), **95**, 661 (1909) *57*

I_2O_5

CO **III-2608**

When carbon monoxide is passed over iodine pentoxide at a temperature of 150 to 200°C, this reaction changes the carbon monoxide to carbon dioxide.

$$I_2O_5 + 5CO \rightarrow I_2 + 5CO_2$$

A. H. Gill and E. H. Bartlett, J. Ind. Eng. Chem., **2**, 9 (1910)
Ref., M. C. Teague, ibid., **12**, 964 (1920) *22*
Ref., T. E. Thrope, J. Chem. Soc. (London), **83**, 318 (1903) *102*

I_2O_5

CO **III-2609**

Iodine pentoxide decomposes at elevated temperature in a current of carbon monoxide to give iodine and carbon dioxide:

$$I_2O_5 + 5CO \rightarrow I_2 + 5CO_2$$

E. Goutal, Ann. chim. anal., **15**, 1 (1910) *76*

I_2O_5 **III-2610**

CO

Iodine pentoxide reacts with carbon monoxide at 65–75°.

$$5CO + I_2O_5 \rightarrow 5CO_2 + I_2$$

J. E. G., Am. Chem. J., **25**, 345 (1901) *1*
Ref., Lewis and Lacey, ibid., **37**, 1979 (1915)

I_2O_5 **III-2611**

CO

H_2

Carbon monoxide is oxidized when passed over iodine pentoxide at 90–100°C. The reaction is quantitative and may be used for the determination of carbon monoxide. If the mixture contains hydrogen, some of the iodine reacts with the hydrogen. At higher temperatures, the iodine pentoxide oxidizes the hydrogen.

(a) $$5CO + I_2O_5 \rightarrow I_2 + 5CO_2$$

(b) $$H_2 + I_2 \rightarrow 2HI$$

(c) $$5H_2 + I_2O_5 \rightarrow 5H_2O + I_2$$

J. I. Graham, and T. F. Winmill, J. Chem. Soc. (London), **105**, 1996 (1914) *57*

I_2O_5 **III-2612**

C_2H_2

Iodine pentoxide reacts with acetylene.

$$I_2O_5 + C_2H_2 \rightarrow I_2 + 2CO_2 + H_2O$$

A. Gautier, Compt. rend., **142**, 15 (1906) *25*

I_2O_5 **III-2613**

Cs_2CO_3

Iodic anhydride is added to a solution of cesium carbonate, precipitating cesium iodate.

$$I_2O_5 + Cs_2CO_3 \rightarrow \underline{2CsIO_3} + (CO_2)$$

Wheeler, Am. J. Sci., **44**, 123 (1902)
Ref., T. V. Barker, J. Chem. Soc. (London), **93**, 15 (1908) *57*

I_2O_5

NH_3 **III-2614**

Iodine pentoxide is warmed with ammonia.

$$3I_2O_5 + 10NH_3 \rightarrow 5N_2 + 3I_2 + 15H_2O$$

A. Gautier, Compt. rend., **142**, 15 (1906) *25*

I_2O_5

Rb_2CO_3 **III-2615**

Iodic anhydride is added to a solution of rubidium carbonate, precipitating rubidium iodate.

$$I_2O_5 + Rb_2CO_3 \rightarrow \underline{2RbIO_3} + (CO_2)$$

Wheeler, Am. J. Sci., **44**, 123 (1902)
Ref., T. V. Barker, J. Chem. Soc. (London), **93**, 15 (1908) *57*

I_2O_5

SO_3 **III-2616**

Five grams iodine pentoxide and 20 g. sulfur trioxide are heated in a closed tube for 24 hours. The product is dried in a stream of carbon dioxide at 90–100°. A pale yellow solid is formed.

$$I_2O_5 + 2SO_3 \rightarrow I_2O_5 \cdot 2SO_3$$

M. M. P. Muir, J. Chem. Soc. (London), **95**, 662 (1909) *57*

I_2O_5

Zn **III-2617**

Iodates are reduced to iodides when rubbed with zinc powder.

$$I_2O_5 + 6Zn \rightarrow ZnI_2 + 5ZnO$$

Merck, Pharm. Ztg., **50**, 1022
Ref., J. Am. Chem. Soc., **28**, 1750 (1906) *1*

I_2O_5

III-2618

Δ

When iodine pentoxide is heated above 300°, in vacuo, or in a current of dry air, iodine and oxygen are partially liberated, but appreciable quantities of these products remain firmly occluded in the oxide.

$$2I_2O_5 \rightarrow 2I_2 + 5O_2$$

M. Guichard, Compt. rend., **147**, 1307 (1908) *38*

I_2S_2

III-2619

Hg

In order to trap mercury vapor, it is allowed to react with iodine disulfide, forming mercuric iodide.

$$Hg + I_2S_2 \rightarrow HgI_2 + (2S)$$

W. Crookes, Trans. Roy. Soc. (London), **176**, 693 (1885) *105*

$KH(IO_3)_2$

III-2620

KBr

H_2SO_4

Chlorides may be separated from bromides by adding to a mixture of them, sulfuric acid and hydrogen potassium iodate and distilling the bromine and iodine that is liberated.

$$KH(IO_3)_2 + 10KBr + 11H_2SO_4 \rightarrow 11KHSO_4 + 5Br_2 + I_2 + 6H_2O$$

Bugarezky, Z. anorg. Chem., **10**, 387 (1895)
L. W. Andrews, J. Am. Chem. Soc., **29**, 276 (1907) *1*

KI

III-2621

Br_2

Dry powdered potassium iodide exposed to bromine, combines to form potassium bromoiodide. The solid, when heated, loses iodine bromide to form potassium bromide.

(a) $$KI + Br_2 \rightarrow KIBr_2$$

(b) $$KIBr_2 \rightarrow KBr + IBr$$

W. N. Rae, J. Chem. Soc. (London), **107**, 1290 (1915) *57*

KI

Br_2 **III-2622**

$Na_2S_2O_3$

The addition of an excess (1 ml. is sufficient) of potassium iodide solution to free bromine liberates free iodine which may be measured by titration with sodium thiosulfate solution to a starch end point.

(a) $$3Br_2 + 6KI \rightarrow 6KBr + 3I_2$$

(b) $$3I_2 + 6Na_2S_2O_3 \rightarrow 6NaI + 3Na_2S_4O_6$$

E. W. Koenig, Anal. Ed., **11**, 532 (1939) *44*

KI

$C_2H_4Br_2$ **III-2623**

Potassium iodide reacts with α,β-dibromoethylene when the latter is dissolved in methanol, at 75°C according to either of the two following equations.

(a) $$C_2H_4Br_2 + 2KI \rightarrow C_2H_4 + 2KBr + I_2$$

(b) $$C_2H_4Br_2 + 3KI \rightarrow C_2H_4 + 2KBr + KI_3$$

M. W. Brenner and G. L. Poland, Anal. Ed., **10**, 528 (1938) *44*

KI

$CaOCl_2$ **III-2624**

HCl

$C_6H_4(OH)_2$

Triiodoresorcinol is obtained by the interaction of potassium iodide and resorcinol in the presence of calcium hypochlorite and hydrochloric acid.

$$6CaOCl_2 + 12KI + 12HCl + 2C_6H_4(OH)_2 \rightarrow 6CaCl_2 + 6HI + 12KCl + 2C_6HI_3(OH)_2 + 6H_2O$$

Dagener, J. f. prakt. Chem., **20:2**, 324 (1879)
Ref., V. Cofman, J. Chem. Soc. (London), **115:2**, 1041 (1919) *48*

KI

III-2625

$CuSO_4$

Under ordinary conditions copper sulfate reacts with potassium iodide to give cuprous iodide, potassium sulfate and iodine.

$$2CuSO_4 + 4KI \rightarrow 2CuI + 2K_2SO_4 + I_2$$

E. Pannain, Gazz. chim. ital., **38**[1], 353 (1908) *21*

KI

III-2626

HNO_2

An iodide in solution may be oxidized to free iodine with nitrous acid (in presence of a mineral acid.)

$$2KI + 2HNO_2 + H_2SO_4 \rightarrow I_2 + 2NO + K_2SO_4 + 2H_2O$$

G. N. Lewis and M. Randall, Thermodynamics, New York
Ref., J. F. McClendon, Physiol. Rev., **7**, 241 (1927) *1*

KI

III-2627

H_2O_2

$FeSO_4$

In the presence of a dilute solution of ferrous sulfate, the reaction between potassium iodide and hydrogen peroxide is very sensitive, for a starch indicator turns blue immediately. Probably ferrous iodide is formed first and then decomposed.

(a) $$FeSO_4 + 2KI \rightarrow FeI_2 + K_2SO_4$$

(b) $$FeI_2 + H_2O_2 \rightarrow Fe(OH)_2 + I_2$$

C. Weltzien, Compt. rend., **62**, 642 (1866) *29*

KI

III-2628

H_2O_2

Potassium iodide and hydrogen peroxide react to form potassium dioxide and hydriodic acid.

$$2KI + H_2O_2 \rightarrow K_2O_2 + 2HI$$

M. C. Weltzien, Chem. News, **13**, 160 (1866) *101*

KI

H_2O_2 **III-2629**

When hydrogen peroxide is added to a neutral solution of potassium iodide, the liquid immediately becomes alkaline. Iodine separates and oxygen is given off. The reaction appears to take place in three steps.

(a) $$2KI + H_2O_2 \rightarrow K_2O_2 + 2HI$$

(b) $$K_2O_2 + H_2O \rightarrow 2KOH + O$$

(c) $$2HI + O \rightarrow H_2O + I_2$$

C. Weltzien, Compt. rend., **62**, 642 (1866) *29*

KI

H_2O_2 **III-2630**

Iodine separates when an aqueous solution of potassium iodide is treated with hydrogen peroxide.

(a) $$2KI + H_2O_2 \rightarrow 2HI + K_2O_2$$

(b) $$2K_2O_2 + 2H_2O \rightarrow 4KOH + O_2$$

(c) $$O_2 + 4HI \rightarrow 2H_2O + I_2$$

E. Schöne, Ann., **195**, 243 (1879) *20*

KI

H_2SO_4 **III-2631**

An excess of concentrated sulfuric acid is added to potassium iodide.

$$2KI + 3H_2SO_4 \rightarrow 2KHSO_4 + 2H_2O + SO_2 + I_2$$

H. Jackson, J. Chem. Soc. (London), **43**, 339 (1883) *25*

KI

$HgCl_2$ **III-2632**

When potassium iodide is treated with an excess of mercuric chloride, the double salt first formed decomposes with the precipitation of red mercuric iodide.

(a) $$HgCl_2 + 4KI \rightarrow 2KCl + HgI_2 \cdot 2KI$$

(b) $$HgCl_2 + HgI_2 \cdot 2KI \rightarrow 2KCl + \underline{2HgI_2}$$

O. Kaspar, Analyst, **6**, 203 (1881) *40*

KI

III-2633

KIO_3

HCl

Hydrochloric acid acts on a mixture of potassium iodide and potassium iodate to liberate free iodine.

$$6HCl + 5KI + KIO_3 \rightarrow 6KCl + 3H_2O + 3I_2$$

M. Blanchetiere, Bull. soc. chim. (France) [4], **19**, 304 (1916) *31*

KI

III-2634

KIO_3

H_2SiF_6

Fluosilicic acid is decomposed by a mixture of potassium iodate and iodide.

$$5KI + H_2SiF_6 + KIO_3 \rightarrow 3K_2F_2 + \underline{SiO_2} + H_2O + 3I_2$$

A. Hileman, Am. J. Sci. [4], **22**, 383 (1906) *25*

KI

III-2635

$KMnO_4$

The determination of iodine in the presence of chlorine and bromine involves the following reactions:

(a) $KI + 2KMnO_4 + H_2O \rightarrow KIO_3 + 2KOH + 2MnO_2$

(b) $KIO_3 + 5KI + 6HCl \rightarrow 6KCl + 3H_2O + 3I_2$

(c) $3I_2 + 6Na_2S_2O_3 \rightarrow 6NaI + 3Na_2S_4O_6$

Baughman and Skinner, J. Ind. Eng. Chem., **11**, 563 (1919)
Ref., J. W. Sale, J.A.O.A.C., **4**, 380 (1921) *42*

KI

III-2636

$KMnO_4$

Potassium iodide is boiled with a solution of potassium permanganate.

$$3KI + 6KMnO_4 + 3H_2O \rightarrow 3KIO_3 + 6MnO_2 + 6KOH$$

Groeger, Z. angew. Chem., **13**, 52 (1894) *25*

KI

KOBr **III-2637**

In the estimation of anions, the use of hypobromite gave results 2–4% higher than the theoretical. This error is avoided by the use of excess bromide. Kolthoff and Stenger* have shown that errors may be avoided by the use of calcium hypochlorite with excess KBr. Under these conditions, "hypobromite in statu nascendi" is also formed, which immediately reacts with oxidizable substances. Direct titration is possible using potentiometric methods.
The addition of a known amount of arsenite (which must be corrected for) induces the following reaction with iodides:

$$KI + 3KOBr \rightarrow KIO_3 + 3KBr$$

O. Tomíček and P. Filipovič, Coll. Czech. Com., **10**, 340 (1938) *87*

KI

K_2CrO_4 **III-2638**

HCl

By operating at a cold temp. potassium chromate reacts with potassium iodide and hydrochloric acid to give potassium chloride, chromic chloride, water and iodine.

$$2K_2CrO_4 + 6KI + 16HCl \rightarrow 10KCl + 2CrCl_3 + 8H_2O + 6I$$

Gino Gallo, Gazz. chim. ital., **37**[1], 459 (1907) *21*

KI

$K_2Cr_2O_7$ **III-2639**

SO_3

Pure iodine may be obtained by treating potassium iodide with potassium dichromate and sulfur trioxide.

$$(6KI + K_2Cr_2O_7 + 7SO_3 \rightarrow 3I_2 + Cr_2(SO_4)_3 + 4K_2SO_4)$$

M. Luchs, Chem. News, **6**, 171 (1862) *101*

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* I. M. Kolthoff and V. A. Stenger, Anal. Ed., **7**, 79 (1935)

KI

III-2640

$(NH_4)_2SO_4$

Ammonium sulfate is transformed into ammonium iodide on the addition of potassium iodide.

$$((NH_4)_2SO_4 + 2KI \rightarrow K_2SO_4 + 2NH_4I)$$

Jacobsen, Chem. News, **9**, 169 (1864) *101*

KI

III-2641

O_2

In presence of light, solutions of iodides are altered by oxygen, according to the equations:

(a) $$4I^- + 4H^+ + O_2 \rightarrow 2I_2 + 2H_2O$$

(b) $$I_2 + I^- \rightarrow I_3^-$$

G. Jones, J. Phys. Chem., **34**, 673 (1930)
Ref., L. de Brouckère, J. Chim. phys., **27**, 543 (1930) *69*

KI

III-2642

O_3

Ozone oxidizes potassium iodide in solution.

$$O_3 + 2KI + H_2O \rightarrow O_2 + I_2 + 2KOH$$

C. Harries, Z. Elektrochem., **17**, 632 (1911) *86*

KI

III-2643

O_3

HCl

For the quantitative determination of small amounts of ozone the ozone is absorbed by an acidified solution of potassium iodide.

$$2KI + O_3 + 2HCl \rightarrow O_2 + I_2 + 2KCl + H_2O$$

W. E. Vaughan and W. A. Noyes, Jr., J. Am. Chem. Soc., **52**, 560 (1930) *16*

KI

ε **III-2644**

The electrolysis of potassium iodide solutions in acetone yields iodine at the anode.

$$(2KI \rightarrow I_2 + 2K)$$

Lasczynski, J. Russ, Phys. Chem. Soc., **32**, 797 (1900)
Ref. E. H. Archibald and L. A. Piguet, Trans. Roy. Soc. (Canada) [III], **11**, 107 (1917) *69*

KI

ε **III-2645**

If electrodes are introduced into a solution in which ferric chloride is being reduced by potassium iodide, the iodide, and not the ferric salt, is reduced.

$$KI + H_2O \rightarrow KOH + HI$$

D. A. Welsh, Trans. Roy. Soc. (Canada) [III], **7**, 209 (1913) *69*

KIO_3

Cl_2 **III-2646**

(KOH)

Chlorine is passed into a hot, strongly alkaline solution of potassium iodate. The periodate separates in tetragonal bipyramids.

$$KIO_3 + Cl_2 + (2KOH) \rightarrow \underline{KIO_4} + (2KCl + H_2O)$$

Rammelsberg, Ann. Phys. Chem., **134**, 368 (1868)
Ref., T. V. Barker, J. Chem. Soc. (London), **93**, 15 (1908) *57*

KIO_3

III-2647

HF

By evaporating a solution of potassium iodate in 40% hydrofluoric acid, crystals of potassium difluoperiodate are obtained. The same type reaction occurs with sodium, cesium, rubidium and ammonium iodates.

(a) $KIO_3 + 2HF \rightarrow \underline{KIO_2F_2} + H_2O$

(b) $NaIO_3 + 2HF \rightarrow \underline{NaIO_2F_2} + H_2O$

(c) $CsIO_3 + 2HF \rightarrow \underline{CsIO_2F_2} + H_2O$

(d) $RbIO_3 + 2HF \rightarrow \underline{RbIO_2F_2} + H_2O$

(e) $NH_4IO_3 + 2HF \rightarrow \underline{NH_4IO_2F_2} + H_2O$

R. F. Weinland and O. Lauenstein, Z. anorg. Chem., **20**, 30 (1899) *28*

KIO_3

III-2648

H_3SbO_3

HCl

At a low range of acidities (2.4–3.5*N* acid) there is a definite endpoint in the reduction of iodate to iodine by antimonites. In the presence of tartrate the acidity may be as low as 1*N* (a). At acidities of 4–6*N* the iodine liberated in (a) is oxidized to iodine monochloride (b).

(a) $2KIO_3 + 5H_3SbO_3 + 2HCl \rightarrow I_2 + 5H_3SbO_4 + 2KCl + H_2O$

(b) $2I_2 + KIO_3 + 6HCl \rightarrow 5ICl + KCl + 3H_2O$

N. H. Furman and C. O. Miller, J. Am. Chem. Soc., **59**, 159 (1937) *16*

KIO_3

III-2649

KI

HCl

The following reaction has been used as a source of radioactive iodine in the introduction of iodine into aniline and insulin.

$$KIO_3 + 5KI + 6HCl \rightarrow 3I_2 + 3H_2O + 6KCl$$

I. Reiner, A. S. Keston and M. Green, Science, **96**, 362 (1942) *44*

KIO_3

KI **III-2650**

H_2SO_4

When under normal temp. potassium iodate and potassium iodide are allowed to react with dilute sulfuric acid, iodine is extracted from both iodide and iodate.

$$KIO_3 + 5KI + 6H_2SO_4 \rightarrow 6KHSO_4 + 3H_2O + 3I_2$$

M. Spica, Gazz. chim. ital., **24**[1], 91 (1894) *21*

KIO_3

MoO_3 **III-2651**

A solution of one part of potassium periodate and one part of molybdenum trioxide is boiled for some time. Crystals of the double compound separate.

$$2KIO_3 + 2MoO_3 \rightarrow K_2OI_2O_5 \cdot 2MoO_3$$

A. Rosenheim and O. Liebknecht, Ann., **308**, 49 (1899) *20*

KIO_4

KI **III-2652**

With excess periodate present potassium iodide and periodate form potassium iodate.

$$3KIO_4 + KI \rightarrow 4KIO_3$$

H. H. Willard and L. H. Greathouse, J. Am. Chem. Soc., **60**, 2869 (1938) *16*

KIO_4

KI **III-2653**

H_3BO_3

In the formation of iodate from excess periodate and iodide, the excess of periodate may be determined by titrating with standard arsenite the iodine liberated from iodide in neutral solution.

$$KIO_4 + 2KI + 4H_3BO_3 \rightarrow KIO_3 + I_2 + K_2B_4O_7 + 6H_2O$$

H. H. Willard and L. H. Greathouse, J. Am. Chem. Soc., **60**, 2869 (1938) *16*

$NH_3 \cdot NI_3$

III-2654

HCl

Finely powdered iodine is digested for several minutes with NH_4OH (sp. g. 0.90) and the excess ammonium hydroxide drawn off. The brown mixture of iodine and nitrogen iodide is washed with water and several cc. of conc. HCl added. A sharp crackling noise indicates a violent reaction. Excess HCl is used to carry the iodine monochloride which is formed into solution.

$$NH_3 \cdot NI_3 + 5HCl \rightarrow 3ICl + 2NH_4Cl$$

L. B. Howell and W. A. Noyes, J. Am. Chem. Soc., **42**, 1009 (1920) *16*

$NH_4H_2(IO_3)_3$

III-2655

$Na_2S_2O_3$

Sodium tetrathionate is formed when ammonium tri-iodate is titrated with sodium thiosulfate.

$$3NH_4H_2(IO_3)_3 + 6Na_2S_2O_3 \rightarrow 3Na_2S_4O_6 + 5NaIO_3 + NaI + 3H_2O + 3NH_4IO_3$$

Riegler, Bull. assoc. chimist., **24**, 528
Ref., J. Am. Chem. Soc., **30**, 427 (1908) *1*

NH_4I

III-2656

I_2

Solubility results in the NH_4I-iodine-benzene and toluene systems show that NH_4I_3 is the only binary periodide which forms above 6°C. This behavior is entirely unlike that of KI which does form a ternary solvated compound but no binary iodide.

$$NH_4I + I_2 \rightarrow NH_4I_3$$

H. W. Foote and W. Bradley, J. Phys. Chem., **37**, 31 (1933) *85*

NH_4I

III-2657

Na

The presence of ammonium iodide in liquid ammonia may be readily detected by sodium which will liberate hydrogen.

$$2NH_4I + 2Na \rightarrow 2NaI + 2NH_3 + H_2$$

W. C. Johnson, G. H. Morey, and A. E. Kott, J. Am. Chem. Soc., **54**, 4280 (1932) *16*

NaI

$(CH_3)_2CO$ **III-2658**

A crystalline compound $NaI \cdot 3(CH_3)_2CO$ is produced by dissolving anhydrous sodium iodide in hot acetone, and then cooling to room temperature. The compound is decomposed by heating.

(a) $NaI + 3(CH_3)_2CO \rightarrow NaI \cdot 3(CH_3)_2CO$

(b) $NaI \cdot 3(CH_3)_2CO \rightarrow NaI + 3(CH_3)_2CO$

K. Shipsey and E. Werner, J. Chem. Soc. (London), **103**, 1255 (1913) *57*

NaI

$COCl_2$ **III-2659**

Phosgene, free from acid gases, may be absorbed in saturated (1.9 per cent) acetone solution of sodium iodide.

$$COCl_2 + 2NaI \rightarrow CO + 2NaCl + I_2$$

M. P. Matuszak, Anal. Ed., **6**, 457 (1934) *44*

NaI

$Ce(SO_4)_2$ **III-2660**

HCl

The oxidation of iodide ion by ceric sulfate has been shown to proceed as follows.

$$2NaI + 4Ce(SO_4)_2 + 2HCl \rightarrow 2Ce_2(SO_4)_3 + Na_2SO_4 + 2ICl + H_2SO_4$$

D. T. Englis and A. A. Knoepfelmacher, Anal. Ed., **17**, 393 (1945) *44*

NaI

$Ce(SO_4)_2$ **III-2661**

H_2O

Sodium iodide may be quantitatively oxidized by ceric sulfate solution in the presence of hydrochloric acid according to the following equation.

$$NaI + 6Ce(SO_4)_2 + 3H_2O \rightarrow NaIO_3 + 3Ce_2(SO_4)_3 + 3H_2SO_4$$

W. W. Hilty and D. T. Wilson, Anal. Ed., **11**, 637 (1939) *44*

NaI

III-2662

$(CH_3)_2CO$

Pure acetone may be prepared by dissolving anhydrous sodium iodide in the acetone solution by gentle warming. The solution so prepared is cooled to ordinary temperature, or better to $-8°$ by an ice-salt mixture. The crystalline product, $NaI \cdot 3C_2H_6O$, separates out, and is filtered and washed. The pure acetone is obtained from this by heating and collecting the acetone.

(a) $NaI + 3(CH_3)_2CO \rightarrow NaI \cdot 3(CH_3)_2CO$

(b) $NaI \cdot 3(CH_3)_2CO \rightarrow NaI + 3(CH_3)_2CO$

Kathleen Shipsey and E. A. Werner, J. Chem. Soc. (London), **103**, 1255 (1913) *25*

NaI

III-2663

(O)

A colloidal solution of iodine, stabilized with gum arabic, may be formed by rapidly acidifying a cold dilute solution of sodium iodide and sodium iodate (proportion of five to one). A more stable solution is formed under similar conditions by employing hypoiodous acid, freshly prepared. (Treat a 1–2% solution of NaOH with iodine until a red color forms. Decant from excess iodine.)

(a) $2HCl + NaI + NaIO_3 \rightarrow 2NaCl + HI + HIO_3$

(b) $5HI + HIO_3 \rightarrow 3I_2 + 3H_2O$

(c) $2NaOH + I_2 \rightarrow Na_2I(OI) + H_2O$

(d) $Na_2I(OI) + 2HCl \rightarrow 2NaCl + HI + HOI$

(e) $HI + HOI \rightarrow I_2 + H_2O$

W. L. Chandler and E. S. Miller, J. Phys. Chem., **31**, 1092 (1927) *85*

$NaIO_3$

III-2664

MoO_3

A solution of one part of sodium iodate and one part molybdenum trioxide is boiled for a long time. The boiling solution is filtered and from the filtrate colorless crystals separate. These are the double compound of sodium periodate with molybdenum trioxide. $Na_2OI_2O_5 \cdot 2MoO_3$.

$$2NaIO_3 + 2MoO_3 \rightarrow Na_2OI_2O_5 \cdot 2MoO_3$$

A. Rosenheim and O. Liebknecht, Ann., **308**, 49 (1899) *20*

$NaIO_4$

$Ba(NO_3)_2$ **III-2665**

H_2O

Sodium periodate is treated with a solution of barium nitrate.

$$2NaIO_4 + 2Ba(NO_3)_2 + H_2O \rightarrow \underline{Ba_2I_2O_9} + 2NaNO_3 + 2HNO_3$$

C. F. Rammelsberg, Pogg. Ann., **134**, 391 (1868) *25*

NaOI

NaOCl **III-2666**

NaOH

Sodium hypoiodite quickly decomposes according to equations, (a) and (b).

(a) $$3NaOI \rightarrow 2NaI + NaIO_3$$

(b) $$NaOI + 3NaOCl + NaOH + H_2O \rightarrow 3NaCl + H_3Na_2IO_6$$

W. M. Dehn, J. Am. Chem. Soc., **33**, 1599 (1911) *1*

NaOI

$Na_2S_2O_3$ **III-2667**

Iodine in alkaline solution forms hypoiodate which reacts with sodium thiosulfate giving iodide and sulfate.

$$(2Na_2S_2O_3 + 4NaOI + 2NaOH \rightarrow 3Na_2SO_4 + 4NaI + H_2S)$$

J. P. Batey, Analyst, **36**, 132 (1911) *40*

$Na_2H_3IO_6$

$AgNO_3$ **III-2668**

An aqueous suspension of sodium periodate is shaken with a neutral aqueous solution of silver nitrate for a long time. A precipitate of silver paraperiodate forms.

$$Na_2H_3IO_6 + 3AgNO_3 \rightarrow \underline{Ag_3IO_5} + 2NaNO_3 + HNO_3 + H_2O$$

O. Rosenheim and O. Liebknecht, Ann., **308**, 57 (1899) *20*

$Na_2H_3IO_6$

III-2669

H_2O_2

Sodium dimesoperiodate reacts with hydrogen peroxide.

$$Na_2H_3IO_6 + H_2O_2 \rightarrow NaIO_3 + NaOH + 2H_2O + O_2$$

V. Auger, Compt. rend., **153**, 1005 (1911) *25*

$Na_2H_3IO_6$

III-2670

I_2

Sodium dimesoperiodate reacts with iodine.

$$2Na_2H_3IO_6 + I_2 \rightarrow 3NaIO_3 + NaI + 3H_2O$$

E. Pecard, Compt. rend., **130**, 1705 (1900) *25*

$5Na_2OI_2O_7 \cdot 12WO_3$

III-2671

KCl

An aqueous solution of pentasodium hexatungstate periodate treated with potassium gives the potassium hexatungstate periodate. This salt crystallizes with eight H_2O.

A similar reaction occurs when this reagent is treated with strontium chloride or barium chloride. The strontium salt crystallizes with twenty eight H_2O and the barium salt with twelve H_2O.

$$5Na_2OI_2O_7 \cdot 12WO_3 + 10KCl \rightarrow 5K_2OI_2O_7 \cdot 12WO_3 + 10NaCl$$

A. Rosenheim and O. Liebknecht, Ann., **308**, 61 (1899) *20*

$Na_3H_2IO_6$

III-2672

$CH_2OH \cdot CHOH \cdot CH_2OH$

H_2SO_4

A typical oxidation of an organic compound using periodic acid may be illustrated by citing the determination of glycerol. The reactions are as follows:

(a) $$CH_2OH \cdot CHOH \cdot CH_2OH + 2Na_3H_2IO_6 + 2H_2SO_4 \rightarrow 2HCHO + HCOOH + 2NaIO_3 + 2Na_2SO_4 + 5H_2O$$

(b) $$Na_3H_2IO_6 + Na_3AsO_3 + 2NaHCO_3 \rightarrow Na_3AsO_4 + NaIO_3 + 2Na_2CO_3 + 2H_2O$$

G. F. Smith and F. R. Duke, Anal. Ed., **15**, 120 (1943) *44*

$Na_4I_2O_9$

$Sr(NO_3)_2$ **III-2673**

Sodium dimesoperiodate reacts with strontium nitrate in dilute ammoniacal solution.

$$2Na_4I_2O_9 + 3Sr(NO_3)_2 \rightarrow Sr_3(IO_5)_2 + 2NaIO_4 + 6NaNO_3$$

A. Ihre, Ber., **3**, 316 (1870) *25*

RbI

Br_2 **III-2674**

Dry powdered rubidium iodide, exposed to bromine, combines to form rubidium bromo-iodide. The solid, when heated, liberates iodine bromide.

(a) $$RbI + Br_2 \rightarrow RbIBr_2$$

(b) $$RbIBr_2 \rightarrow RbBr + IBr$$

W. N. Rae, J. Chem. Soc. (London), **107**, 1290 (1915) *57*

RbI

Cl_2 **III-2675**

RbOH

Chlorine is passed into a hot, concentrated solution of rubidium iodide and hydroxide, whereby the sparingly soluble iodate is precipitated.

$$RbI + 6RbOH + 3Cl_2 \rightarrow \underline{RbIO_3} + (6RbCl + 3H_2O)$$

T. V. Barker, J. Chem. Soc. (London), **93**, 15 (1908) *57*

RbI

I_2 **III-2676**

At 6° and 25°C the binary compound RbI_3 is the only stable solid in the I_2-RbI-toluene system. Two ternary polyiodides, $RbI \cdot 6I \cdot 4C_6H_6$ and $RbI \cdot 7I \cdot 4C_6H_6$ are stable in the I_2-RbI-benzene system.

(a) $$RbI + I_2 \rightarrow RbI_3$$

(b) $$RbI + 3I_2 + 4C_6H_6 \rightarrow RbI \cdot 6I \cdot 4C_6H_6$$

(c) $$RbI + 3\tfrac{1}{2}I_2 + 4C_6H_6 \rightarrow RbI \cdot 7I \cdot 4C_6H_6$$

H. W. Foote and M. Fleischer, J. Phys. Chem., **44**, 640 (1940) *85*

$RbIO_3$

III-2677

Cl_2
(KOH)

Rubidium iodate is oxidized to the periodate by passing chlorine into hot, strongly alkaline solution of rubidium iodate.

$$RbIO_3 + Cl_2 + (2KOH) \rightarrow \underline{RbIO_4} + (2KCl + H_2O)$$

T. V. Barker, J. Chem. Soc. (London), **93**, 16 (1908) *57*

$RbIO_3 \cdot HIO_3$

III-2678

HF

By cooling off a heated solution of acid rubidium iodate in 40% hydrofluoric acid, crystals of rubidium tetrafluorodiiodate are obtained.

$$RbIO_3 \cdot HIO_3 + 4HF \rightarrow \underline{RbIO_2 \cdot 2F_2 \cdot HIO_2 \cdot 2H_2O}$$

R. F. Weinland and O. Köppen, Z. anorg. Chem., **22**, 256 (1900) *28*

SnI_4

III-2679

KNH_2

A solution of stannic iodide in liquid ammonia reacts with potassium-imide.

$$SnI_4 + 6KNH_2 \rightarrow 4KI + K_2Sn(NH_2)_6$$

Fitzgerald, J. Am. Chem. Soc., **29**, 1693 (1907) *25*

SnI_4

III-2680

KOH

Stannic iodide reacts with a solution of potassium hydroxide.

$$SnI_4 + 6KOH \rightarrow 4KI + K_2Sn(OH)_6$$

Fitzgerald, J. Am. Chem. Soc., **29**, 1693 (1907) *25*

$UO_2(IO_3)_2$

III-2681

KCl

Uranyl iodate is decomposed by a boiling solution of potassium chloride.

$$2UO_2(IO_3)_2 + 2KCl \rightarrow KIO_3 + UO_2Cl_2 + UO_2K(IO_3)_3$$

P. Artmann, Z. anorg. Chem., **80**, 280 (1913) *25*

Ag_2IrCl_6

HCl **III-2682**

Δ

The free acid is obtained when silver hexachloroiridate is treated with hydrochloric acid. On heating this, tetrachloroiridious acid is produced.

(a) $Ag_2IrCl_6 + 2HCl \rightarrow H_2IrCl_6 + (2AgCl)$

(b) $H_2IrCl_6 \rightarrow HIrCl_4 + (HCl + Cl)$

Duffour, Compt. rend., **152**, 1393, 1591
Ref., J. L. Howe, J. Am. Chem. Soc., **34**, 160 (1912) *1*

$Ag_3Ir(C_2O_4)_3$

HCl **III-2683**

The free acid is formed when the silver salt of tri-oxalatoiridious acid is treated with hydrochloric acid. This, by a reversible reaction goes over into diaquodioxalatoiridous acid.

(a) $Ag_3Ir(C_2O_4)_3 + 3HCl \rightarrow H_3Ir(C_2O_4)_3 + (3AgCl)$

(b) $H_3Ir(C_2O_4)_3 + 2H_2O \rightarrow HIr(H_2O)_2 \cdot (C_2O_4)_2 + H_2C_2O_4$

Duffour, Compt. rend., **152**, 1393, 1591
Ref., J. L. Howe, J. Am. Chem. Soc., **34**, 160 (1912) *1*

$Cu_3Ir_2(CN)_{12}$

NH_3 **III-2684**

H_2O

Blue colored crystals are formed when a solution of ammonia is added to copper iridium cyanide.

$$Cu_3Ir_2(CN)_{12} + 6NH_3 + 4H_2O \rightarrow \underline{Ir_2(CN)_{12} \cdot Cu_3(NH_3)_6 \cdot 4H_2O}$$

E. Rimbach and F. Korten, Z. anorg. Chem., **52**, 406 (1907) *28*

Ir

III-2685

Cl

The reactions by which metallic iridium is changed to a soluble chloride may be represented by a single equation

$$Ir + 4Cl + 2Cl^- \rightarrow IrCl_6^{=}$$

E. Wichers, W. G. Schlecht, and C. L. Gordon, B. S., J. Res., **33**, 366 (1944) *16*

Ir

III-2686

Cl_2

Iridium hexachloride is synthesized by passing chlorine over Ir metal at 360–400°C. It is a greenish yellow solid with a melting point of approximately 30°C.

$$Ir + 3Cl_2 \rightarrow IrCl_6$$

O. Ruff and J. Fischer, Z. Elektrochem., **33**, 560 (1927) *86*

Ir

III-2687

Cl_2

Iridium tetrachloride is formed by passing chlorine over iridium mixed with sodium or potassium chloride at low red heat.

$$Ir + 2Cl_2 \rightarrow IrCl_4$$

A. Gutbier, Z. physik. Chem., **69**, 304 (1909)
A. Gutbier and M. Riess, Ref., Ber., **42**, 3905, 4770 (1909)
Ref., J. L. Howe, J. Am. Chem. Soc., **33**, 187 (1911) *1*

Ir

III-2688

$(KF)_3HF \cdot PbF_4$

A mixture of potassium and lead fluoiridates is formed when finely powdered iridium is heated with the double salt of lead tetrafluoride.

$$2((KF)_3HF \cdot PbF_4) + 5Ir + 7H_2F_2 \rightarrow 3K_2IrF_6 + 2PbIrF_6 + (8H_2)$$

H. I. Schlesinger and M. W. Tapley, J. Am. Chem. Soc., **46**, 281 (1924) *1*

Ir

NaCl **III-2689**

Cl_2

Ammonium chloroiridate is prepared by heating in a combustion tube a mixture of sodium chloride and iridium black and passing in chlorine, (a). The sodium chloride formed is dissolved and treated with ammonium chloride and chlorine (b).

(a) $(Ir + 2NaCl + 2Cl_2 \rightarrow Na_2IrCl_6)$

(b) $(Na_2IrCl_6 + 2NH_4Cl \rightarrow (NH_4)_2IrCl_6 + 2NaCl)$

A. Gutbier, Ber., **42**, 3905 (1909)
Ref., E. H. Archibald and J. W. Kern, Trans. Roy. Soc. (Canada) [III], **11**, 7 (1917) *69*

$IrCl_3$

NH_4OH **III-2690**

When iridium trichloride is treated with boiling concentrated ammonium hydroxide at least three ammoniacal compounds are formed: a tri- (difficulty soluble), a tetra-, and a penta- (easily soluble) ammonium iridium chloride.

(a) $IrCl_3 + 3NH_4OH \rightarrow Ir(NH_3)_3Cl_3 + (3H_2O)$

(b) $IrCl_3 + 4NH_4OH \rightarrow Ir(NH_3)_4Cl_3 + (4H_2O)$

(c) $IrCl_3 + 5NH_4OH \rightarrow Ir(NH_3)_5Cl_3 + (5H_2O)$

J. Landin, Sv. Kem. Tidsk., **I**, 67 (1889) *10*

$IrCl_3$

$NaBrO_3$ **III-2691**

When sodium bromate is added to hot trivalent iridium ions, the iridium is oxidized to the quadrivalent form.

$$6IrCl_3 + 6HCl + NaBrO_3 \rightarrow 6IrCl_4 + (NaBr + 3H_2O)$$

Gilchrist, B. S. J. Res., **12**, 294 (1934) *93*

$IrCl_3$

III-2692

$NaHCO_3$

When trivalent iridium ions are treated with sodium hydrogen carbonate, a flocculent, gelatinous precipitate is formed.

$$2IrCl_3 + 6NaHCO_3 \rightarrow \underline{Ir_2O_3 \cdot H_2O} + (6NaCl + 6CO_2 + 2H_2O)$$

Gilchrist, B. S. J. Res., **12**, 295 (1934) *98*

$IrCl_3$

III-2693

$NaNO_2$

NH_4Cl

Iridium can be separated from platinum by precipitating the double ammonium nitrate (a).
The mother liquor is heated with hydrochloric acid and the platinum precipitated, (b).

(a) $(IrCl_3 + 4NaNO_2 + NH_4Cl \rightarrow \underline{NH_4Ir(NO_2)_4} + 4NaCl)$

(b) $Pt(NO_2)_2 + 4HCl + 2NH_4Cl \rightarrow (NH_4)_2PtCl_6 + 2H_2O + 2NO)$

Leider, Compt. rend., **131**, 888 (1900)
Ref., E. H. Archibald and J. W. Kern, Trans. Roy. Soc. (Canada) [III], **11**, 7 (1917) *69*

$IrCl_4$

III-2694

$C_5H_5N \cdot HCl$

$C_6H_5NH_2 \cdot HCl$

A solution of iridium tetrachloride will react with pyridine hydrochloride. Black needles will be obtained. The same type reaction occurs with aniline hydrochloride.

(a) $IrCl_4 + 2C_5H_5N \cdot HCl \rightarrow \underline{IrCl_4 \cdot (2C_5H_5N \cdot HCl)}$

(b) $IrCl_4 + 2C_6H_5NH_2 \cdot HCl \rightarrow \underline{IrCl_4 \cdot (2C_6H_5NH_2 \cdot HCl)}$

E. Rimbach and F. Korten, Z. anorg. Chem., **52**, 345 (1907) *28*

$IrCl_4$

H_2S **III-2695**

Hydrogen sulfide will precipitate iridium disulfide in solutions which contain 20% concentrated hydrochloric acid by volume. The complete precipitation requires treatment with hydrogen sulfide for 2–3 hours, the solution being kept just below the boiling point, while a fairly rapid stream of hydrogen sulfide is being passed into it.

$$IrCl_4 + 2H_2S \rightarrow \underline{IrS_2} + (4HCl)$$

Hillebrand and Lundell, Applied Inorganic Analysis, 276 (1929) *93*

$IrCl_4$

KNO_2 **III-2696**

If a solution of iridium tetrachloride is warmed with potassium nitrite, an olive-green coloration is produced. If, however, the solution is boiled with an excess of KNO_2 for some time, it becomes yellow, and a part of the iridium settles out as a yellowish-white powder, difficultly soluble in cold hydrogen chloride or hot water.

(a) $$IrCl_4 + KNO_2 \rightarrow IrCl_3 + KCl + NO_2$$

(b) $$2IrCl_3 + 6KNO_2 \rightarrow \underline{K_3[Ir(NO_2)_6] \cdot K_3[IrCl_6]}$$

Treadwell and Hall, Analytical Chemistry, 5th ed., **1**, 569 *93*

$IrCl_4$

NaOH **III-2697**

Sodium hydroxide added to a solution of iridium tetrachloride or sodium chloroiridate changes the color from dark red to green. Warming changes it to azure blue.

$$2IrCl_4 + 2NaOH \rightarrow 2IrCl_3 + NaCl + NaOCl + H_2O$$

Treadwell and Hall, Analytical Chemistry, 5th. Ed., **1**, 569 *93*

$IrCl_4$

NaOH **III-2698**

Cl_2

When chlorine is passed into a hot, alkaline solution of iridium tetrachloride, iridium tetrahydroxide is precipitated.

$$IrCl_4 + 6NaOH + Cl_2 \rightarrow \underline{Ir(OH)_4} + (NaOCl + 5NaCl + H_2O)$$

Gilchrist, B. S. J. Res., **12**, 284 (1934) *93*

$IrCl_4$

$NaHCO_3$ **III-2699**

When sodium acid carbonate is added to a boiling solution of quadrivalent iridium ions, the deep green hydrated oxide, which is quite granular, is formed and precipitated. The reaction is complete at pH of 4–6.

$$IrCl_4 + 4NaHCO_3 \rightarrow \underline{IrO_2 \cdot 2H_2O} + (4NaCl + 4CO_2)$$

Gilchrist, B. S. J. Res., **12**, 295 (1934) *93*

$IrCl_4$

RbCl **III-2700**

By mixing together a solution of iridium tetrachloride with a solution of rubidium chloride in excess, a deep red precipitate will be obtained.

$$IrCl_4 + 2RbCl \rightarrow \underline{IrRb_2Cl_6}$$

E. Rimbach and F. Korten, Z. anorg. Chem., **52**, 406 (1907) *28*

$IrCl_4$

Zn **III-2701**

Iridium can be separated from platinum by precipitation from an acid solution of iridic chloride by means of zinc (a). The precipitate is treated with aqua regia and the residue is converted into potassium chloroiridate (b).

(a) $$(IrCl_4 + 2Zn \rightarrow 2ZnCl_2 + Ir)$$

(b) $$(Ir + 4HCl + 2KCl \rightarrow K_2IrCl_6 + 2H_2)$$

U. Anthony, Gazz. chim. ital., **22**, 275
Ref., E. H. Archibald and J. R. Kern, Trans. Roy. Soc. (Canada), [III], **11**, 7 (1917) *69*

$Ir(NH_3)_5Br(NO_2)_2$

H_2SO_4 **III-2702**

When iridiumpentamminobromonitrite is treated with sulfuric acid, the nitrous acid evolved, alcohol added, and the precipitated bromosulfate recrystallized from water; yellow, lustrous tablets of monohydrated iridiumpentamminobromosulfate are formed.

$$Ir(NH_3)_5Br(NO_2)_2 + H_2SO_4 + H_2O \rightarrow (2HNO_2) + Ir(NH_3)_5BrSO_4 \cdot H_2O$$

W. Palmaer, K. Sven. Vet. Ak. Handl. Öfvers., **48**, No. 6, 417 (1891) *10*

$Ir(NH_3)_5Br_3$

$AgNO_2$ **III-2703**

Solutions of iridium pentamminotribromide and silver nitrite are mixed, the silver bromide filtered off, and the filtrate yields light-yellow, thick, well-formed prisms of anhydrous iridiumpentamminobromonitrite.

$$Ir(NH_3)_5Br_3 + 2AgNO_2 \rightarrow \underline{2AgBr} + Ir(NH_3)_5Br(NO_2)_2$$

W. Palmaer, K. Sven. Vet. Ak. Handl. Öfvers., **48**, No. 6, 417 (1891) *10*

$Ir(NH_3)_5Cl(NO_3)_2$

H_2SiF_6 **III-2704**

Mixed solutions of iridiumpentamminochloronitrate and fluosilicic acid yield an immediate white scale-like precipitate of iridiumpentamminochlorofluosilicate.

$$Ir(NH_3)_5Cl(NO_3)_2 + H_2SiF_6 \rightarrow (2HNO_3) + \underline{Ir(NH_3)_5ClSiF_6}$$

W. Palmaer, K. Sven. Vet. Ak. Handl. Öfvers., **46**, No. 10, 368 (1889) *10*

$Ir(NH_3)_5Cl(NO_3)_2$

KI **III-2705**

When solutions of iridiumpentamminochloronitrate and potassium iodide are mixed iridiumpentamminochloroiodide precipitates as an immediate, whitish-yellow, crystalline powder.

$$Ir(NH_3)_5Cl(NO_3)_2 + 2KI \rightarrow \underline{Ir(NH_3)_5ClI_2} + (2KNO_3)$$

W. Palmaer, K. Sven. Vet. Ak. Handl. Öfvers., **46**, No. 10, 368 (1889) *10*

$Ir(NH_3)_5Cl(NO_3)_2$

III-2706

$K_4Fe(CN)_6$

Mixed solutions of iridiumpentamminochloronitrate and potassium ferrocyanide yield slowly light-yellow, feathery crystals of iridiumpentamminochloroferrocyanide.

$$2Ir(NH_3)_5Cl(NO_3)_2 + K_4Fe(CN)_6 \rightarrow (4KNO_3) + (Ir(NH_3)_5Cl)_2 \cdot Fe(CN)_6$$

W. Palmaer, K. Sven. Vet. Ak. Handl. Öfvers., **46**, No. 10, 368 (1889) *10*

$Ir(NH_3)_5Cl(NO_3)_2$

III-2707

$(NH_4)_2C_2O_4$

Solutions of iridiumpentamminochloronitrate and ammonium oxalate react and yield fine white needles of anhydrous iridiumpentamminochlorooxalate.

$$Ir(NH_3)_5Cl(NO_3)_2 + (NH_4)_2C_2O_4 \rightarrow (2NH_4NO_3) + Ir(NH_3)_5ClC_2O_4$$

W. Palmaer, K. Sven. Vet. Ak. Handl. Öfvers., **46**, No. 10, 367 (1889) *10*

$Ir(NH_3)_5ClSO_4$

III-2708

$BaBr_2$

Solutions of iridiumpentamminochlorosulfate and barium bromide are mixed, the barium sulfate filtered off, and the filtrate yields light-yellow, rhombic crystals of anhydrous iridiumpentamminochlorobromide.

$$Ir(NH_3)_5ClSO_4 + BaBr_2 \rightarrow Ir(NH_3)_5ClBr_2 + \underline{BaSO_4}$$

W. Palmaer, K. Sven. Vet. Ak. Handl. Öfvers., **46**, No. 10, 364 (1889) *10*

$Ir(NH_3)_5ClSO_4$

$BaCO_3$ **III-2709**

HI

A solution of iridiumpentamminochlorosulfate is mixed with barium carbonate and hydriodic acid, the barium sulfate filtered off, and the filtrate yields yellowish brown rhombic crystals of anhydrous iridiumpentamminochloroiodide.

$$Ir(NH_3)_5ClSO_4 + BaCO_3 + 2HI \rightarrow \underline{BaSO_4} + Ir(NH_3)_5ClI_2 + (CO_2 + H_2O)$$

W. Palmaer, K. Sven. Vet. Ak. Handl. Öfvers., **46**, No. 10, 365 (1889) *10*

$Ir(NH_3)_5ClSO_4$

$BaCl_2$ **III-2710**

Solutions of iridiumpentamminochlorosulfate and barium chloride are mixed, the barium sulfate filtered off, and the filtrate yields light-yellow, octahedral crystals of anhydrous iridiumpentamminotrichloride.

$$Ir(NH_3)_5ClSO_4 + BaCl_2 \rightarrow \underline{BaSO_4} + Ir(NH_3)_5Cl_3$$

W. Palmaer, K. Sven. Vet. Ak. Handl. Öfvers., **46**, No. 6, 361 (1889) *10*

$Ir(NH_3)_5ClSO_4$

$Ba(NO_3)_2$ **III-2711**

Solutions of iridiumpentamminochlorosulfate and barium nitrate are mixed, the barium sulfate filtered off, and the filtrate yields clusters of whitish-yellow, rhombic crystals of anhydrous iridiumpentamminochloronitrate.

$$Ir(NH_3)_5ClSO_4 + Ba(NO_3)_2 \rightarrow Ir(NH_3)_5Cl(NO_3)_2 + \underline{BaSO_4}$$

W. Palmaer, K. Sven. Vet. Ak. Handl. Öfvers., **46**, No. 10, 366 (1889) *10*

$Ir(NH_3)_5Cl_3$

III-2712

$AgNO_2$

Solutions of iridiumpentamminotrichloride and silver nitrite are mixed, the silver chloride filtered off, and the filtrate yields light-yellow, well-formed, prisms of anhydrous iridiumpentamminochloronitrite.

$$Ir(NH_3)_5Cl_3 + 2AgNO_2 \rightarrow Ir(NH_3)_5Cl(NO_2)_2 + \underline{2AgCl}$$

W. Palmaer, K. Sven. Vet. Ak. Handl. Öfvers., **46**, No. 10, 366 (1889) *10*

$Ir(NH_3)_5Cl_3$

III-2713

H_2SO_4

C_2H_5OH

Iridiumpentamminotrichloride is treated with concentrated sulfuric acid and alcohol added to precipitate the chlorosulfate, which on recrystallization from water yields long, light-yellow prisms of di-hydrated iridium pentamminochlorosulfate.

$$Ir(NH_3)_5Cl_3 + H_2SO_4 + 2H_2O \rightarrow \underline{Ir(NH_3)_5ClSO_4 \cdot 2(H_2O)} + (2HCl)$$

W. Palmaer, K. Sven. Vet. Ak. Handl. Öfvers., **46**, No. 10, 362 (1889) *10*

$Ir(NH_3)_5Cl_3$

III-2714

KOH

Solutions of iridiumpentamminotrichloride and potassium hydroxide yield when boiled iridiumpentamminotrihydroxide.

$$Ir(NH_3)_5Cl_3 + 3KOH \rightarrow Ir(NH_3)_5 \cdot (OH)_3 + (3KCl)$$

W. Palmaer, K. Sven. Vet. Ak. Handl. Öfvers., **48**, No. 6, 424 (1891) *10*

$Ir(NH_3)_5Cl_3$

III-2715

$PtCl_4$

When solutions of iridiumpentamminotrichloride and of platinum tetrachloride are mixed iridiumpentamminochloroplatinate precipitates as a heavy, orange-yellow powder made up of small, rectangular tablets.

$$Ir(NH_3)_5Cl_3 + PtCl_4 \rightarrow \underline{Ir(NH_3)_5Cl_3 \cdot PtCl_4}$$

W. Palmaer, K. Sven. Vet. Ak. Handl. Öfvers., **46**, No. 10, 367 (1889) *10*

$Ir(NH_3)_5Cl_3$

Δ **III-2716**

Iridiumpentamminotrichloride, when heated, breaks down into iridium, ammonium chloride and ammonia.

$$2Ir(NH_3)_5Cl_3 + \Delta \rightarrow 2Ir + 6NH_4Cl + 2NH_3 + (N_2)$$

W. Palmaer, K. Sven. Vet. Ak. Handl. Öfvers., **46**, No. 10, 361 (1889) *10*

$Ir(NH_3)_5 \cdot H_2O \cdot (NO_3)_3$

Δ **III-2717**

Iridiumaquopentamminotrinitrate, when heated yields iridiumpentamminotrinitrate.

$$Ir(NH_3)_5 \cdot H_2O \cdot (NO_3)_3 + \Delta \rightarrow (H_2O) + Ir(NH_3)_5(NO_3)_3$$

W. Palmaer, K. Sven. Vet. Ak. Handl. Öfvers., **48**, No. 6, 425 (1891) *10*

$Ir(NH_3)_5(OH)_3$

HNO_3 **III-2718**

Iridiumpentamminotrihydroxide, treated with nitric acid yields rhombic crystals of iridiumaquopentamminotrinitrate.

$$Ir(NH_3)_5(OH)_3 + 3HNO_3 \rightarrow (2H_2O) + Ir(NH_3)_5 \cdot H_2O \cdot (NO_3)_3$$

W. Palmaer, K. Sven. Vet. Ak. Handl. Öfvers., **48**, No. 6, 424 (1891) *10*

Ir_2O_3

H_2SO_4 **III-2719**

When iridium oxide is dissolved in dilute sulfuric acid and the solution allowed to crystallize in a vacuum, iridium sulfate is obtained.

$$Ir_2O_3 + 3H_2SO_4 \rightarrow \underline{Ir_2(SO_4)_3} + 3H_2O$$

L. Marino, Z. anorg. Chem., **42**, 213 (1904) *28*

Ir_2O_3

III-2720

Δ

Analysis of the temperature-pressure curve for the thermal dissociation of iridium oxide shows that this process takes place in two steps.

(a) $$2Ir_2O_3 \rightleftharpoons 3IrO_2 + Ir$$

(b) $$IrO_2 \rightleftharpoons Ir + O_2$$

L. Wöhler and W. Witzmann, Z. Elektrochem., **14**, 103 (1908) *86*

$Ir_2(SO_4)_3$

III-2721

Cs_2SO_4

A solution of cesium sulfate is added to a solution of iridium sulfate. On concentrating and cooling the solution, crystals of iridium-cesium alum are formed. The same type reaction occurs with rubidium sulfate, potassium sulfate, ammonium sulfate and thallous sulfate.

$$Ir_2(SO_4)_3 + Cs_2SO_4 + 24H_2O \rightarrow \underline{Ir_2(SO_4)_3 \cdot Cs_2SO_4 \cdot 24H_2O}$$

L. Marino, Z. anorg. Chem., **42**, 213 (1904) *28*

$K \cdot Ir(H_2O)_2(C_2O_4)_2$

III-2722

KOH

A hydroxyaquo salt is formed by the action of potassium hydroxide on the potassium salt of diaquodioxalatoiridious acid.

$$KIr(H_2O)_2(C_2O_4)_2 + KOH \rightarrow K_2Ir(OH)(H_2O)(C_2O_4)_2 + (H_2O)$$

Duffour, Compt. rend., **152**, 1393, 1591
Ref., J. S. Howe, J. Am. Chem. Soc., **34**, 160 (1912) *1*

K_2IrCl_6

III-2723

$H_2C_2O_4$

H_2O

$K_2(H_2O)IrCl_5$ is obtained readily by reduction of the chloroiridates by oxalic acid, evaporating to dryness to expel the HCl formed and to crystallize the salt.

$$2K_2IrCl_6 + H_2C_2O_4 + 2H_2O \rightarrow 2K_2(H_2O)IrCl_5 + 2CO_2 + 2HCl$$

M. M. Delepine, Bull. soc. chim. [4], **3**, 905 (1908) *31*

K_2IrCl_6

$K_2C_2O_4$ **III-2724**

M. Vèzes prepared potassium chloroiridite with remarkable ease and rapidity by reduction of potassium chloroiridate with potassium oxalate dissolved in H_2O and heated on a water bath.

$$2K_2IrCl_6 + K_2C_2O_4 \rightarrow 2K_3IrCl_6 + 2CO_2$$

M. Vezes, Procès verbaux des Séances de la Societé des Sciences physiques et naturelles de Bordeaux, Année 1900-1901, p. 112
Ref., M. M. Delepine, Bull. soc. chim. [4], **3**, 904 (1908) *31*

K_2IrCl_6

KNO_2 **III-2725**

If a solution of the K or NH_4 salt in hot water is treated with KNO_2, then an olive-green solution of the trivalent Ir salt is formed. If, however, the solution is boiled with an excess of KNO_2 for some time, it becomes yellow, and finally a white precipitate is formed.

(a) $$K_2IrCl_6 + KNO_2 \rightarrow K_3IrCl_6 + NO_2$$

(b) $$2K_3IrCl_6 + 6KNO_2 \rightarrow \underline{K_3Ir(NO_2)_6 \cdot K_3IrCl_6} + 6KCl$$

Treadwell and Hall, Analytical Chemistry, 5th ed., **1**, 569 *93*

K_2IrCl_6

HI **III-2726**

Chloroiridates oxidize hydriodic acid liberating iodine and forming chloroiridites.

$$K_2IrCl_6 + KI \rightarrow K_3IrCl_6 + I$$

M. Delepine, Bull. soc. chim. [4], **5**, 1085 (1909) *31*

K_3IrCl_6

H_2O **III-2727**

The potassium and ammonium salts of type M_3IrCl_6 dissolved in water form a solution which, on evaporation, yields a mixture according to the following equation:

$$K_3IrCl_6 + H_2O \rightarrow K_2(H_2O)IrCl_5 + KCl$$

M. M. Delepine, Bull. soc. chim. [4], **3**, 905 (1908) *31*

K_3IrCl_6

III-2728

$K_2C_2O_4$

Potassium iridodichloroxalate is obtained by double decomposition between the chloroiridite and potassium oxalate. Solutions of the salts are mixed, heated and crystallization allowed to take place.

$$K_3IrCl_6 + 2K_2C_2O_4 \rightarrow K_3IrCl_2(C_2O_4)_2 + 4KCl$$

Vezes and Duffour, Bull. soc. chim. [4], **5**, 869 (1909) *31*

K_3IrCl_6

III-2729

$K_2C_2O_4$

Racemic potassium iridium oxalate can be prepared by treating potassium hexachloroiridate with a neutral solution of potassium oxalate.

$$(K_3IrCl_6 + 3K_2C_2O_4 \rightarrow K_3Ir(C_2O_4)_3 + 6KCl)$$

Delépine, Bull. soc. chim. [5], **1**, 1256 (1934)
Ref., J. P. Mathieu, J. Chim. phys., **33**, 78 (1936) *69*

$K_3Ir(CN)_6$

III-2730

$AgNO_3$

NH_3

A solution of potassium iridium cyanide will react with a solution of silver nitrate yielding a white precipitate of silver iridium cyanide, which is soluble in ammonia. Colorless crystals will crystallize from this solution.

(a) $K_3Ir(CN)_6 + 3AgNO_3 \rightarrow \underline{Ag_3Ir(CN)_6} + 3KNO_3$

(b) $Ag_3Ir(CN)_6 + 2NH_3 + 3H_2O \rightarrow \underline{Ir(CN)_6Ag_3(NH_3)_2 \cdot 3H_2O}$

E. Rimbacher and F. Korten, Z. anorg. Chem., **52**, 345 (1907) *28*

Li_2IrCl_6

III-2731

$Li_2C_2O_4$

Lithium chloroiridate reacts readily with lithium oxalate when heated, yielding large, black, rhombohedral, deliquescent crystals.

$$2Li_2IrCl_6 + Li_2C_2O_4 \rightarrow 2Li_3IrCl_6 + 2CO_2$$

M. Delepine, Bull. soc. chim. [4], **15**, 508 (1915) *31*

$(NH_4)_3IrCl_6$

H_2SO_4 **III-2732**

At 100° sulfuric acid will react with a concentrated solution of ammonium chloroiridite according to the following equation:

$$2(NH_4)_3IrCl_6 + 2H_2SO_4 \rightarrow 2(NH_4)_2IrCl_6 + (NH_4)_2SO_4 + SO_2 + 2H_2O$$

M. Delépine, Bull. soc. chim. [4], **9**, 835 (1911) *31*

$Na_2(IrCl_6)$

H_2S **III-2733**

Hydrogen sulfide passed through a solution of sodium chloroiridate will at first decolorize the solution, and then cause a deposition of iridium trisulfide, which is readily soluble in ammonium polysulfide.

(a) $2Na_2(IrCl_6) + H_2S \rightarrow 2IrCl_3 + \underline{S} + (4NaCl + 2HCl)$

(b) $2IrCl_3 + 3H_2S \rightarrow \underline{Ir_2S_3} + (6HCl)$

Treadwell and Hall, Analytical Chemistry, 5th Ed., **1**, 569 *93*

$Na_2(IrCl_6)$

KNO_2 **III-2734**

A solution of one part disodiumhexachloroiridate is treated with 2–3 parts of potassium nitrite. The precipitate which forms is dissolved in a solution of potassium nitrite and an equal volume of nitrous acid added. The solution is allowed to crystallize and the crystals are recrystallized five to six times from water. Pure potassium dichlorotetranitroiridate forms as bright yellow crystals.

$$Na_2(IrCl_6) + 5KNO_2 \rightarrow [IrCl_2(NO_2)_4]K_3 + 2NaCl + 2KCl + NO_2$$

A. Werner and O. de Vries, Ann., **364**, 86 (1909) *20*

Na_2IrCl_6

KOH **III-2735**

O

Oxygen is passed into a concentrated hot solution of sodium iridium chloride, which was made alkaline with potassium hydroxide solution. Iridium dioxide will be formed.

(a) $2Na_2IrCl_6 + 2NaCl + 2KOH \rightarrow 2Na_3IrCl_6 + KOCl + KCl + H_2O$

(b) $2Na_3IrCl_6 + 6KOH \rightarrow Ir_2O_3 + 3H_2O + 6KCl + 6NaCl$

(c) $Ir_2O_3 + O \rightarrow 2IrO_2$

L. Wöhler and W. Witzmann, Z. anorg. Chem., **57**, 323 (1908) *28*

$Na_2(IrCl_6)$

NH_4Cl **III-2736**

Ammonium chloride precipitates dark-red ammonium chloroiridate, which is insoluble in a saturated solution of ammonium chloride.

$$Na_2(IrCl_6) + 2NH_4Cl \rightarrow \underline{(NH_4)_2(IrCl_6)} + (2NaCl)$$

Treadwell and Hall, Analytical Chemistry, 5th Ed., **1**, 569 *93*

$Na_2(IrCl_6)$

$NaNO_2$ **III-2737**

5 grams of sodiumhexachloroiridate are dissolved in water and the solution warmed to 80–90°C. 40 grams of sodium nitrite are added in small portions. A copious evolution of nitric oxide occurs and the color of the solution changes from dark to a dirty brown and finally to a reddish brown color. The solution is heated to just below boiling for 15 minutes and then allowed to cool. The solution is concentrated over sulfuric acid and yellowish crystals separate. These are crystallized from water and lemon yellow crystals of sodium dichlorotetranitroiridate with two H_2O separate.

$$Na_2(IrCl_6) + 5NaNO_2 \rightarrow [IrCl_2(NO_2)_4]Na_3 + 4NaCl + NO_2$$

A. Werner and O. de Vries, Ann., **364**, 81 (1909) *20*

Na_2IrCl_6

$Na_2C_2O_4$ **III-2738**

Oxalatotetrachloroiridite is formed when sodium hexachloroiridate is treated with sodium oxalate.

$$2Na_2IrCl_6 + 2Na_2C_2O_4 \rightarrow 2Na_3IrCl_4(C_2O_4) + (2NaCl + Cl_2)$$

Duffour, Compt. rend., **152**, 1393–1591
J. L. Howe, J. Am. Chem. Soc., **34**, 160 (1912) *1*

$Na_3IrCl_2(NO_2)_4$

NaI **III-2739**

$C_2N_2H_8 \cdot H_2O$

Triethylene diamine iridium chloride and some diethylene diamine iridium dinitro iodide are obtained when iridium III sodium dichlorotetranitrite is heated in a sealed tube with ethylene diamine hydrate and then, treated with sodium iodide.

$$(2Na_3IrCl_2(NO_2)_4 + 5C_2N_2H_8 \cdot H_2O + 4NaI \rightarrow Ir(C_2N_2H_8)_3I_3 + Ir(C_2N_2H_8)_2(NO_2)_2I + 4NaCl + 6NaNO_2 + 5H_2O)$$

J. P. Mathieu, J. Chim. phys., **33**, 78 (1936) *69*

Na_3IrCl_6

$NaNO_2$ **III-2740**

Sodium nitroiridite can be prepared in three steps from solutions of the chloroiridite and sodium nitrite.

(a) $2Na_3IrCl_6 + 6Ba(OH)_2 \rightarrow Ba_3[Ir(OH)_6]_2 + 3BaCl_2 + 6NaCl$

(b) $Ba_3[Ir(OH)_6]_2 + 6H_2SO_4 \rightarrow Ir_2(SO_4)_3 + \underline{3BaSO_4} + 12H_2O$

(c) $Ir_2(SO_4)_3 + 12NaNO_2 \rightarrow 2Na_3Ir(NO_2)_6 + 3Na_2SO_4$

Mathieu and Cornevin, J. Chim. phys., **36**, 271 (1939) *69*

Na_3IrCl_6

$NaNO_2$ **III-2741**

One mole of sodium hexachloroiridate heated with four moles of sodium nitrite gives iridium III sodium dichlorotetranitrite.

$$(Na_3IrCl_6 + 4NaNO_2 \rightarrow Na_3IrCl_2(NO_2)_4 + 4NaCl)$$

J. P. Mathieu, J. Chim. phys., **33**, 78 (1936) *69*

$[Ir(NO_2)_2(NH_3)_4]Cl$

III-2742

$AgNO_2$

2 grams of dinitrotetramminoiridium chloride are digested with 0.8 gram of silver nitrite in water on the water bath until no more silver chloride is formed. The residue is filtered off and the solution allowed to cool. Colorless crystals of dinitrotetramminoiridium nitrite with one H_2O are formed.

$$[Ir(NO_2)_2(NH_3)_4]Cl + AgNO_2 \rightarrow [Ir(NO_2)_2(NH_3)_4]NO_2 + AgCl$$

A. Werner and O. de Vries, Ann., **364**, 100 (1909) *20*

$[IrCl_2(NH_3)_4]Cl$

III-2743

HBr

A warm aqueous solution of dichlorotetramminoiridium monochloride is treated with concentrated hydrobromic acid. A yellow gelatinous mass precipitates. It is crystallized from warm water containing hydrobromic acid and yellow needles of dichlorotetramminoiridium monobromide with one H_2O are formed.

$$[IrCl_2(NH_3)_4]Cl + HBr \rightarrow [IrCl_2(NH_3)_4]Br + HCl$$

A. Werner and O. de Vries, Ann., **364**, 119 (1909) *20*

$[IrCl_2(NH_3)_4]Cl$

III-2744

H_2SO_4

A warm aqueous solution of dichlorotetramminoiridium monochloride is treated with concentrated sulfuric acid until no more hydrochloric acid is evolved. As the solution cools, yellow crystals of dichlorotetramminoiridium sulfate with one H_2O separate.

$$2[IrCl_2(NH_3)_4]Cl + H_2SO_4 \rightarrow [IrCl_2(NH_3)_4]_2SO_4 + 2HCl$$

A. Werner and O. de Vries, Ann., **364**, 118 (1909) *20*

$[Ir(NO_2)_2(NH_3)_4]Cl$

KBr **III-2745**

A warm solution of dinitrotetramminoiridium chloride is treated with excess potassium bromide and crystals of dinitrotetramminoiridium bromide with one H_2O form. These are crystallized from water containing either potassium bromide or hydrobromic acid and colorless crystals are obtained.

$$[Ir(NO_2)_2(NH_3)_4]Cl + KBr \rightarrow [Ir(NO_2)_2(NH_3)_4]Br + KCl$$

A. Werner and O. de Vries, Ann., **364**, 97 (1909) *20*

$[IrCl_2(NH_3)_4]Cl$

KI **III-2746**

A warm aqueous solution of dichlorotetramminoiridium monochloride is treated with solid potassium iodide and the difficultly soluble dichlorotetramminoiridium monoiodide precipitates. It is crystallized from water containing potassium iodide.

$$[IrCl_2(NH_3)_4]Cl + KI \rightarrow [IrCl_2(NH_3)_4]I + KCl$$

A. Werner and O. de Vries, Ann., **364**, 120 (1909) *20*

$[Ir(NO_2)_2(NH_3)_4]Cl$

KI **III-2747**

A warm aqueous solution of dinitrotetramminoiridium chloride is treated with excess potassium iodide. As the solution cools, a precipitate forms. It is crystallized from water containing a small amount of potassium iodide and needles of dinitrotetramminoiridium iodide form.

$$[Ir(NO_2)_2(NH_3)_4]Cl + KI \rightarrow [Ir(NO_2)_2(NH_3)_4]I + KCl$$

A. Werner and O. de Vries, Ann., **364**, 99 (1909) *20*

$[Ir(NO_2)_2(NH_3)_4]Cl$

III-2748

K_2SO_4

A warm aqueous solution of dinitrotetramminoiridium chloride is treated with potassium sulfate and the precipitate crystallized from water, very slightly acidified with sulfuric acid. White prisms of dinitrotetramminoiridium sulfate containing one half H_2O separate.

$$2[Ir(NO_2)_2(NH_3)_4]Cl + K_2SO_4 \rightarrow [Ir(NO_2)_2(NH_3)_4]_2SO_4 + 2KCl$$

A. Werner and O. de Vries, Ann., **364**, 99 (1909) *20*

$[Ir(NH_3)_4(NO_2)_2]Cl$

III-2749

NH_4Cl

HCl

1 gram of dinitrotetramminoiridium monochloride is mixed with 1 gram of ammonium chloride and 15 cc of concentrated hydrochloric acid and heated in a sealed tube for 9½ hours at 140°C. The resulting solution is filtered and concentrated to a small volume. A yellow precipitate forms which is crystallized from warm water slightly acidified with hydrochloric acid. Yellow needles of dichlorotetramminoiridium monochloride with one H_2O form.

$$[Ir(NH_3)_4(NO_2)_2]Cl + 2HCl \rightarrow [Ir(NH_3)_4Cl_2]Cl + 2HNO_2$$

A. Werner and O. de Vries, Ann., **364**, 115 (1909) *20*

$[Ir(NH_3)_5NO_2]Cl_2$

III-2750

KI

A warm aqueous solution of nitropentamminoiridium dichloride is treated with a solution of potassium iodide and a yellowish brown precipitate forms which is crystallized from warm water. Yellowish brown crystals of nitropentamminoiridium diiodide form.

$$[Ir(NH_3)_5NO_2]Cl_2 + 2KI \rightarrow [Ir(NH_3)_5NO_2]I_2 + 2KCl$$

A. Werner and O. de Vries, Ann., **364**, 105 (1909) *20*

$[Ir(NH_3)_5NO_2]Cl_2$

Na_2SO_4 **III-2751**

A warm concentrated aqueous solution of nitropentamminoiridium dichloride is treated with a solution of sodium sulfate. As the solution cools white needles of nitropentamminoiridium sulfate crystallize.

$$[Ir(NH_3)_5NO_2]Cl_2 + Na_2SO_4 \rightarrow [Ir(NH_3)_5NO_2]SO_4 + 2NaCl$$

A. Werner and O. de Vries, Ann., **364**, 105 (1909) *20*

$[Ir(NH_3)_5H_2O]Cl_3$

$AgNO_2$ **III-2752**

1 gram of aquopentamminoiridium chloride is dissolved in water and an excess of silver nitrite added. The solution is stirred on the water bath until no more silver chloride is formed. The residue is removed by filtration and the filtrate evaporated to dryness on the water bath and the residue heated for two hours. The aquopentamminonitrite is converted to nitropentamminonitrite. The residue is dissolved in hot water, dilute hydrochloric acid added and the solution heated on the water bath until no more nitric oxide is evolved. The solution is filtered to remove the excess silver as chloride and concentrated hydrochloric acid added to the filtrate. A white precipitate forms which is crystallized from warm water acidified with hydrochloric acid. Small colorless crystals of nitropentamminoiridium chloride separate.

(a) $[Ir(NH_3)_5H_2O]Cl_3 + 3AgNO_2$
$\rightarrow [Ir(NH_3)_5NO_2](NO_2)_2 + \underline{3AgCl} + H_2O$

(b) $[Ir(NH_3)_5NO_2](NO_2)_2 + 2HCl \rightarrow [Ir(NH_3)_5NO_2]Cl_2 + 2HNO_2$

A. Werner and O. de Vries, Ann., **364**, 102 (1909) *20*

$K_2[IrCl_6]$

KCN **III-2753**

Potassium cyanoiridate is prepared by action of potassium cyanide on potassium chloroiridate in solution.

$$(K_2[IrCl_6] + 6KCN \rightarrow K_2[Ir(CN)_6] + 6KCl)$$

Mathieu and Cornevin, J. Chim. phys., **36**, 271 (1939) *66*

$K_3[Ir(C_2O_4)_3]$

III-2754

$AgNO_3$

Silver iridooxalate is prepared from silver nitrate and potassium iridooxalate. The product separates in silky yellow needles, exploding at 145–150°C.

$$3AgNO_3 + K_3[Ir(C_2O_4)_3] + 3H_2O \rightarrow Ag_3[Ir(C_2O_4)_3]\cdot 3H_2O + (3KNO_3)$$

C. Gialdine, Atti R. Accad. Lincei, **16**, ii, 648 (1907)
Ref., T. H. Pope, J. Chem. Soc. (London), **94**, 3 (1908) *57*

$Na_3[IrCl_2(NO_2)_4]$

III-2755

NH_3

1 gram of sodium dichlorotetranitroiridate is heated with 4 cc of 27% ammonia solution for 9 hours at 135–40°C. The reaction product is digested in a solution of sodium potassium carbonate repeatedly until it is free of chlorides. Two to five treatments are necessary. The residue is crystallized from hot water. Crystals of trinitroamminoiridium are obtained.

$$Na_3[(IrCl_2(NO_2)_4)] + 3NH_4OH \rightarrow Ir(NO_2)_3(NH_3)_3 + 2NaCl + NaNO_2 + 3H_2O$$

A. Werner and O. de Vries, Ann., **364**, 91 (1909) *20*

$Na_3[IrCl_2(NO_2)_4]$

III-2756

NH_3

HCl

5 grams of sodiumdichlorotetranitroiridate are added to 20 cc of 27% ammonia and heated for nine hours at 170°C. The reaction products are concentrated and freed of excess ammonia by heating on the water bath. Impure trinitro triamminoiridium precipitates. This is removed by filtration and sodium chloride added to the filtrate. Dinitrotetramminoiridium chloride crystallizes out and is dissolved in water and treated with hydrochloric acid to remove nitrites. The solution is allowed to crystallize and crystals of dinitrotetramminoiridium chloride form as white needles.

$$Na_3[IrCl_2(NO_2)_4)] + 4NH_4OH \rightarrow Ir(NO_2O_2(NH_3)_4Cl + NaCl + 2NaNO_2 + 4H_2O$$

A. Werner and O. de Vries, Ann., **364**, 95 (1909) *20*

$Na_3[IrCl_2(NO_2)_4]$

NH_4Cl **III-2757**

A solution of sodiumdichlorotetranitroiridate is treated with ammonium chloride and the precipitate crystallized from water. Yellow colored crystals of ammoniumdichlorotetranitroiridate with two H_2O are formed.

$$Na_3[IrCl_2(NO_2)_4] + 3NH_4Cl \rightarrow (NH_4)_3[IrCl_2(NO_2)_4] + 3NaCl$$

A. Werner and O. de Vries, Ann., **364**, 87 (1909)

List of Abstractors

The following is a list of abstractors who have contributed reactions to Volume II. Their assigned numbers are printed in italics to the right of every entry in the text. Number 25 represents reactions that were contributed before the system of assigning numbers to abstractors was adopted.

1. Miss Kathryn Huffman, Chemist, Morgantown Coal Testing Laboratory, Morgantown, West Virginia.
3. Prof. Leo K. Yanowski, Department of Chemistry, Fordham College, New York, N. Y.
4. Dr. Moritz Kohn, 536 West 113 St., New York 25, N. Y.
5. Dr. Joseph Z. Schneider, (present address unknown)
6. Mr. Jas. K. Hunt, Public Relations Dept., E. I. du Pont, Wilmington, Del.
7. Prof. Thomas W. Davis, Professor of Physical Chemistry, New York University, New York City, N. Y.
8. Mr. J. Robert Spraul, Technology Center Armour Res. Foundation, Chicago, Ill.
9. Mr. R. J. O'Brien, 27 Walnut St., Rutherford, N. J.
10. Prof. Wilhelm Segerblom, Exeter, N. H. (Deceased)
11. Dr. Virgil Lilly, Biological Chemistry, W. Va. Agr. Experiment Station, Morgantown, W. Va.
13. Mr. Carl Armour, M.S., Clarksburg, W. Va.
14. Mr. W. Jacobson, Antioch College, Yellow Springs, Ohio
15. Mr. B. C. Johnson, Dept. of Animal Husbandry, University of Illinois, Urbana, Ill.
16. Mr. C. H. Briggs, Howard Laboratories, Minneapolis, Minn.
17. Mr. C. M. Hawke, Morgantown, W. Va.
19. Miss Irena Z. Eiger, Dept. of Chemistry, Columbia University, New York, N. Y.
20. Mr. James A. Austin, Chemist, Jensen-Salsbery Laboratories, Kansas City, Mo.
21. Mr. P. S. Potesta, Richwood, W. Va.
22. Mr. Benjamin Tatterson, Morgantown, W. Va.
23. Mr. John Russel, Morgantown, W. Va.
24. Mr. David Posten, Morgantown, W. Va.
25. Anonymous
26. Mr. Carl Leiden, 1003 Carroll St., Boone, Iowa
27. Mr. Richard D. Hoak, Fellow, Mellon Institute, Pittsburgh, Pa.
28. Dr. Walter Wagner, Assistant Professor of Chemistry, University of Detroit, Detroit, Mich.
29. Mr. E. Paul Jones, Research Laboratory, General Chemical Co., Laurel Hill, N. Y.
31. Sister Mary Martinette, B.V.M., Mundelein College, Chicago, Ill.
32. Dr. Loretta E. Fox, Mississippi State College for Women, Columbus, Miss.
33. Dr. Philip J. Elving, Dept. of Chemistry, Purdue University, Lafayette, Ind.
34. Dr. Oskar Horowitz, 235 Lefferts Ave., Brooklyn, N. Y.
35. Mr. John F. Rush, Cambridge Springs, Pa.
38. Miss Janet Searles, E. I. du Pont de Nemours & Co., Inc., Electrochemicals Dept., Wilmington, Del.
40. Prof. W. S. McGuire, Dept. of Chemistry, Northeastern University, Boston, Mass.
42. Mr. W. J. Van Ness, 165-8th Ave., Hawthorn, N. J.
44. Prof. Ralph E. Dunbar, Dean School of Chemical Technology, N. Dak. Agricultural College, Fargo, N. Dak.
47. Mr. H. R. Erickson, 1631 N. 51st St., Seattle, Wash.

48. Mr. Bernard M. Rosenthal, 19 Hillside Court, Berkeley, 4, Calif.
50. Mr. Lester Kuhn, 1051 South Park Ave., Buffalo, N. Y.
53. Dr. Clara L. Deasy, Dept. of Physiological Chemistry, Temple University, Philadelphia, Pa.
57. Prof. J. C. Hackney, Dept. of Chemistry, Purdue University, Lafayette, Ind.
60. Dr. G. M. Kosolapoff, Research Chemist, Monsanto Chemical Co., Dayton, Ohio
61. Dr. H. J. Prebluda, Chemist, U. S. Industrial Chemicals, Baltimore, Md.
63. Mr. P. M. Apfelbaum, College of the the City of N. Y., New York, N. Y.
65. Mr. A. S. Richer, 3999 Dickinson Ave., New York City, N. Y.
67. Dr. G. H. Pritham, Dept. of Chemistry, University of Scranton, Scranton, Pa.
69. Dr. Paul A. Giguère, Dept. of Chemistry, Laval University, Quebec, Can.
73. Mr. W. B. Krabeck, 13 Green St., Concord, N. H.
74. Mr. Frank E. Piech, Box 93, Parlin, N. J.
75. Dr. J. H. Wells, Fellow, Mellon Institute, Pittsburgh, Pa.
76. Mr. Norman S. Landau, 909 S. Third St., Champaign, Ill.
77. Dr. L. E. Cheyney, Dept. of Chemistry, University of Akron, Arkon, Ohio
78. Mr. Richard Geckler, Research Chemist, Standard Oil Co., Whiting, Ind.
81. Mrs. J. T. Cassaday, Ph.D., 100 Strawberry Hill, Stamford, Conn.
82. Mr. Allen G. Ring, Chemist, Mallinckrodt Chemical Works, St. Louis, Mo.
84. Miss Dorothy Franz, General Hospital, Morgantown, W. Va.
85. Prof. Andrew Van Hook, Department of Chemistry, Holy Cross College, Worcester, Mass.
86. Dr. Frederick C. Nachod, Senior Chemist, Sterling-Winthrop Research Institute, Rensselaer, N. Y.
87. Mr. R. B. Bradstreet, Research Chemist, 8 Craig Place, Cranford, N. J.
91. Mr. George N. Cade, Research Chemist, Phillips Petroleum Co., 310 West 13th St., Bartlesville, Okla.
93. Mr. I. A. Rothstein, 1948 East 17th Street, Brooklyn, N. Y.
94. Dr. Burton K. Wasson, Dept. of Chemistry McGill University, Montreal, P. Q., Canada
98. Mr. C. G. Ferrari, General Mills, Minneapolis, Minn.
100. Mr. Lawrence T. Johnson, Morgantown, W. Va.
101. Mr. Herman A. Reda, Research Chemist, 4740 Friendship Ave., Pittsburgh, Pa.
102. Mr. Ralph E. Wilmer, Morgantown, W. Va.
103. Mr. Owen C. Greenwell, Morgantown, W. Va.
105. Mr. W. D. Foster, Chemist, U. S. Bureau of Mines, 4800 Forbes St., Pittsburgh, Pa.
106. Mr. P. I. Bowman, Heyden Chemical Corporation, Garfield, N. J.
109. Mr. D. H. Rubin, 15 Lothian Road, Brighton, Mass.
110. Dr. Maynard S. Raasch, Research Chemist, du Pont Experiment Station, 2515 West 18th St., Wilmington, Del.
111. Mr. W. Merle Robinson, Montezuma, Iowa
112. Mr. Samuel Lee, 512 Rosedale Ave., New York City, N. Y.
114. Mr. J. N. Borglin, 215 West 37th Street, Wilmington, Del.
116. Mr. S. M. Fugiel, Chemist, 3757 N. Octavia Ave., Chicago, Ill.
118. Mr. Clifford A. Hampel, Research Chemist, 15620 Loomis Ave., Harvey, Ill.

Journals Used by Abstractors

Abbreviations used precede the full name of the publication.

Acta Acad. Abo.	Acta Academae Aboensis
Acta Chirurg. Sk.	Acta Chirurgica Skandinavica
Acta Med. Sk.	Acta Medica Skandinavica
Acta Pathol. Micro. Sk.	Acta Pathologica et Microbiologica Skandinavica
Acta Soc. Physiol. Sk.	Acta Societatis Physiologicae Skandinavica
Acta Soc. Sci. fenn.	Acta Societatis Scientiarum Fennicae
Acta Univ. Lund.	Acta Universitatis Lundensis
Acta Univ. Lund. Skrifter	Acta Universitatis Lundensis Skrifter
Afhandl. Norsk. Vid. Ak.	Afhandlinger Norske Videnskaps Akademi
Allgem. Oel u. Fett-Ztg.	Allgemeine Oel und Fett-Zeitung
Am. Cer. Soc.	Same as J. Am. Cer. Soc.
Am. Chem. J.	American Chemical Journal
Am. Dyest. Rep.	American Dyestuff Reporter
Am. Gas Light J.	American Gas Light Journal
Am. J. Sci.	American Journal of Science
An. Asoc. quim. Arg.	Anales de la Asociacion quimica Argentina
An. Soc. Espan. fis. quim.	Anales de la Sociedad Española de fisica y quimica
Analyst	The Analyst
Anal. Ed.	Analytical Edition, Industrial and Engineering Chemistry
Ann.	Annalen der Chemie (Liebig's)
Ann. Chim.	Annales de Chimie
Ann. Chim. Anal.	Annales de Chimie Analytique
Ann. de Chim. Appl.	Annales de Chimie analytique et de chimie appliquée
Ann. Chem. u. Pharm.	Same as Ann.
Ann. chim. phys.	Annales de Chemie et de Physique
Ann. Phys.	Annalen der Physik
Ann. Suppl.	Annalen der Chemie und Pharmacie (vol. 1–8 Supplement)
Apoth. Ztg.	Apotheker Zeitung
Archiv. Med.	Archives de Medicine
Archiv. Pharm.	Archiv der Pharmacie
Ark. Kem. Min. Geol.	Arkiv för Kemi, Mineralogi och Geologi
Ark. Mat. Astr. Fys.	Arkiv för Matematik, Astronomi och Fysik
Atti ac. pont. Lincei	Atti dell'accademia pontifica de Nuovi Lincei
Atti Inst. veneto	Atti dell'Instituto veneto di Scienze lettre ed arti
Avhandl. Fys. Kemi Min.	Avhandlingar i Fysik, Kemi Och Mineralogi
Ber.	Berichte der deutschen chemischen Gesellschaft
Ber. deuts. pharm. Ges.	Berichte der deutschen pharmazeutischen Gesellschaft
Berg u. hütten. Ztg.	Berg und hüttenmannische Zeitung
Berl. Akad. Wien	Berliner Akademie Wien
Bio Chem. J.	Biochemical Journal, The
Bio Chem. Z.	Biochemische Zeitschrift

Boll. Chim. farm.	Bollettino Chimico farmaceutico
Brenn. Chem.	Brennstoff-Chemie
Brit. Pat.	British Patent
B. S. J. Res.	Bureau of Standards, Journal of Research
Bull. Acad. Roy. méd. Belg.	Bulletin dL'academie Royale de medecin (Belgique)
Bull. Acad. Sci. (Petrograd)	Bulletin de L'academie des Sciences (Petrograd)
Bull. Am. Cer. Soc.	Bulletin of the American Ceramic Society
Bull. Am. Inst. Min. Eng.	Bulletin of the American Institute of Mining and Metallurgical Engineering
Bull. assoc. Chimist	Bulletin de l'association des Chimistes de Sucérie et de distillérie
Bull. Chem. Soc. (Japan)	Bulletin of the Chemical Society of Japan
Bull. Geol. Upsala	Bulletin of the Geological Institution of the University of Upsala
Bull. Inst. Phys. & Chem. Res.	Bulletin of the Institute of Physical Chemical Research (Tokyo)
Bull. Nat. Res. C.	Bulletin of the National Research Council
Bull. Sec. Sci. Acad. (Roumaine)	Bullitin de la Section Scientific de l'Academie Roumaine
Bull. soc. chim.	Bulletin de la sociéte chimique de France
Bull. soc. chim. (Belg)	Bulletin de la sociéte chimique de Belgique
Bull. soc. chim. biol.	Bulletin de la sociéte chimique biologique
Bull. soc. chim. (France)	Bulletin de la sociéte chimique de France
Bull. soc. chim. (Rom)	Bulletin de la sociéte chimique de Roumaine
Bull. soc. Mulh.	Bulletin de la sociéte industrielle Mulhause
Bull. Tids. Kjemi Bergvesen	Bulletin Tidskrift Kjemi og Bergvesen
Bur. of Chem.	Bureau of Chemistry, U. S. Department of Agriculture
C.A.	Chemical Abstracts
Can. Chem. J.	Canadian Chemical Journal
Can. Chem. & Met.	Canadian Chemistry and Metallurgy
Can. Inst. Min. Met.	Canadian Institute of Mining and Metallurgy
Can. J. Res.	Canadian Journal of Research
Can. Min. & Met. Bull.	Canadian Mining and Metallurgical Bulletin
Cellulosechemie	Cellulosechemie
Centr. Min.	Centralblatt für Mineralogie, Geologie und Paläontologie (Stuttgart)
Cer. Abstr.	Ceramic Abstracts
Cereal Chem.	Cereal Chemistry
Chaleur et ind.	Chaleur et industrie
Chem. Age (L)	Chemical Age (London)
Chem. Age (N. Y.)	Chemical Age (New York)
Chem. Anal.	Chemische Analyse
Chem. Anal.	Chemist Analyst
Chem. Annalen	Chemische Annalen
Chem. Arch.	Chemisches Archiv
Chem. Engr.	Chemical Engineer
Chem. & Eng. News	Chemical and Engineering News
Chem. Ind.	Chemical Industries

Chem. & Ind.	Chemistry and Industry
Chem. et Ind.	Chemie et Industrie
Chem. J. (Nich.)	Chemical Journal (Nicholson's)
Chem. List.	Chemicky Listy pro vedu a promysi
Chem & Met. Eng.	Chemical and Metallurgical Engineering
Chem. News	Chemical News
Chem. ob.	Chemicky Obzor
Chem. Weekbl.	Chemisches Weekblad
Chem. Zbl. (Chem. Zentr.)	Chemisches Zentralblatt
Chem. Zeit	Chemische Zeitschrift
Chem. Ztg.	Chemische Zeitung
Chim. et Ind.	Chimie et Industrie
Coll. Czech. Com.	Collection of Czechoslovak Chemical Communications
Compt. rend.	Comptes rendus hebdomadaires des séances de l'academie des sciences
Dan. Vid. Sel. Forh.	Danske Videnskabs Selskabets Forhandlinger
Dingler's J.	Dingler's Polytechnisches Journal
D.P. (D.R.P.)	German Patent
Electrochem. Met. Ind.	Electrochemical and Metallurgical Industry
Engineering	Engineering
Eng. Min. J.	Engineering Mining Journal
Entdeck. Chem.	Entdeckungen in der Chemie
F. Pat.	French Patent
Fuel	Fuel in Science and Practice
Gazz. chim. ital.	Gazzetta chimica italiana
G. chim. ind.	Giornal de chimica industriale ed applicata
Geol. Fören. För.	Geologiska Föreningens Förhandlingar
Helv. Chim.	Helvetica chimica Acta
Ind. Chim. (Belg.)	Industrie chimique, Belgue, L'
Ind. Eng. Chem.	Industrial and Engineering Chemistry
Ing. Vet. Akad. Handl.	Ingeniors Vetenskaps Akademien Handlingar
Iron Age	Iron Age
J. Agr. Res.	Journal of Agricultural Research
Jahresb.	Jahresberichte
J. Am. Cer. Soc.	Journal of the American Ceramic Society
J. Am. Chem. Soc.	Journal of the American Chemical Society
J. Am. Pharm. Assn.	Journal of the American Pharmaceutical Association
J. Anal. & Appl. Chem.	Journal of Analytical and Applied Chemistry
J. A. O. A. C.	Journal of the Association of Official Agricultural Chemists
Japan J. Chem.	Japanese Journal of Chemistry
J. Appl. Chem. (USSR)	Journal of Applied Chemistry (U.S.S.R.)

Jber. Fort. Chem.	Jahresberichte über die Fortschritte der Chemie
Jber. Leist. Chem. Tech.	Jahresberichte über die Leistungen der Chemischen Technologie
J. Biochem. (Jap.)	Journal of Biochemistry (Japan)
J. Biol. Chem.	Journal of Biological Chemistry
J. Chem. Educ.	Journal of Chemical Education
J. Chem. Pharm.	Journal der Chemie und Pharmacie
J. Chem. Soc. (London)	Journal of the Chemical Society (London)
J. Chem. Soc. (Japan)	Journal of the Chemical Society (Japan)
J. Chem. Phys.	Journal of Chemical Physics
J. chim. phys.	Journal de chimie physique
J. f. Chem. Phys. Min.	Journal für Chemie, Physik und Mineralogi (Berlin)
J. de phys. de chim.	Journal de physique de chimie
J. Frank. Ins.	Journal of the Franklin Institute
Jenaer Zeit.	Jenaer Zeitschrift
Jern. Ann.	Jernkontorets Annaler (Stockholm)
J. Gen. Chem. (USSR)	Journal of General Chemistry (U.S.S.R.)
J. Ind. Eng. Chem.	Journal of Industrial and Engineering Chemistry (Vols. 1–14 incl.)
J. Org. Chem.	Journal of Organic Chemistry
J. Gasbeleucht.	Journal der Gasbeleuchtung
J. O. S. A.	Journal of the Optical Society of America
J. Pharm.	Journal of Pharmacy
J. Pharm. Chimie	Journal de pharmacie et de chimie
J. Phys. Chem.	Journal of Physical Chemistry
J. Phys. Chem. (USSR)	Journal of Physical Chemistry (U.S.S.R.)
J. Phys. Radium.	Journal de physique et le Radium
J. f. prakt. Chem.	Journal für praktische Chemie
J. & Proc. Austral. Chem. Inst.	Journal and Proceedings of the Australian Chemical Institute
J. Res. B. S.	Journal of Research of the National Bureau of Standards
J. Russ. Phys. Chem. Soc.	Journal of the Russian Physical Chemical Society
J. S. A. Chem. Inst.	Journal of the South African Chemical Institute
J. Soc. Chem. Ind. (London)	Journal of the Society of Chemical Industry (London)
J. Soc. Chem. Ind. (Japan)	Journal of the Society of Chemical Industry (Japan)
J. Soc. Glass Tech.	Journal of the Society of Glass Technology
J. Wash. Acad. Sc.	Journal of the Washington Academy of Sciences
Kemi Min.	Kemi och Mineralogi (Afhandlingar)
Kolloid Chem. Beih.	Kolloid Chemische Beihefte
Kolloid-Zeit.	Kolloid-Zeitschrift
K. Sven. Vet. Ak. Bih	Kungliga Svenska Vetenskaps Akademiens, Bihang
K. Dan. viden. sel skr.	K. Danske videnskabernes selskabs skrifter
K. Sven. Vet. Ak. Handl	Kungliga Svenska Vetenskaps Akademiens Handlingar
K. Sven. Vet. Ak. Över.	Kungliga Svenksa Vetenskaps Akademiens Handlingar, Översigt

L'Orosi	Orosi Bollettino di chimica farmacia e Scienze affini Firenze
Med. K. Vet. Nobelinst.	Meddelanden från Kungliga Vetenskaps Nobelinstitutet
Med. Stat. Prov.	Meddelanden från Statens Provningsanstaltet (Stockholm)
Mem. Am. Acad. Arts Sci.	Memoirs of the American Academy of Arts and Sciences
Mem. Coll. Sci. Eng.	Memoirs of the College of Sciences and Engineering
Met. Chem. Eng.	Metallurgical and Chemical Engineering
Metallurg	Metallurg U.S.S.R.
Mettallurgie	Mettallurgie
Mikrochem.	Mikrochemie
Mineralog. Mag.	Mineralogical magazine
Mod. Plastics	Modern Plastics
Monatsh.	Monatshefte für Chemie
Nature	Nature
Nord. Med. Ark.	Nordiskt Mediciniskt Arkiv
Nov. Acta. Soc. (Upsala)	Nova Acta Regiae Societatis Scientarum Upsaliensis
N. Y. Agr. Exp. Sta.	New York State Agricultural Experiment Station
Off. Gaz. U. S. Pat. Office	Official Gazette of the U. S. Patent Office
Oil & Gas J.	Oil and Gas Journal
Oil & Soap J.	Oil and Soap Journal
Over. K. Dan. Vid. Sel. For.	Oversigt Konglige Danske Videnskabernes Selskabs Forhandlinger
Pharm. J.	Pharmaceutical Journal
Pharm. J. & Trans.	Pharmaceutical Journal and Transactions
Pharm. Mag.	Pharmaceutical Magazine
Pharm. Post	Pharmazeutische Post
Pharm. Soc. (Edin.)	Pharmaceutical Society of Edinburgh
Pharm. Ztg.	Pharmazeutische Zeitung
Pharm. Weekbl.	Pharmaceutische Weekblad
Phil. Mag.	Philosophical Magazine
Phil. Trans.	Philosophical Transactions
Physiol. Rev.	Physiological Review
Plastics	(Same as Modern Plastics)
Pogg. Ann.	Poggendorff's Annalen
Proc. Acad. Sci. Amst.	Proceedings of the Royal Academy of Sciences (Amsterdam)
Proc. Am. Acad.	Proceedings of the American Academy of Arts and Sciences
Proc. Nat. Acad.	Proceedings of the National Academy of Sciences
Proc. Roy. Soc. (London)	Proceedings of the Royal Society of London
Proc. Roy. Soc. Edin.	Proceedings of the Royal Society (Edinburgh)
Proc. S. Af. Ass. Anal. Chem.	Proceedings of the South African Association of Analytical Chemists

Proc. S. Af. Chem. Inst.	Proceedings of the South African Chemical Institute
Proc. Soc. Chem. Ind.	Proceedings of the Society of Chemical Industry (Victoria)
Proc. U. S. Naval Inst.	Proceedings of the U. S. Naval Institute
Quart. J. Ind. Chem. Soc.	Quarterly Journal of the Indian Chemical Society
Rech. Sur l'acid.	Recherches sur l'acide iodique (Paris)
Rec. trav. chim.	Recueil des travaux chimique des Pays-Bas
Rend. Acc. Lincei	Rendiconti della R. Accademia dei Lincei
Rev. chim. Ind.	Revue de chimie Industrielle
Rev. facul. cience.	Revista de la facultad de ciencias Quimicas
Rev. gen. de chim.	Revue generale de chimie pure et applique
Rev. gen. mat. Plast.	Revue general de materie Plastique
Rev. prod. Chim.	Revue des produits chimiques, La
Roczniki Chem.	Roczniki Chemji
Russ. Jahrb. Pharm.	Russiches Jahrbuch der Pharmacie
Science N. S.	Science, New Series
Sitzungsber. K. Akad. Wiss. (Berlin)	Sitzungsberichte K. Akademie der Wissenschaften Berlin
Stahl u. Eisen	Stahl und Eisen
Sv. farm. Tidskr.	Svensk Pharmaceutisk Tidskrift
Sv. foto. Tidskr.	Svensk fotografisk Tidskrift
Sv. kem. Tidskr.	Svensk kemisk Tidskrift
Tids. Kemi Farm. og Ter	Tidskrift Kemi Farmacie og Ter
Tids. Kem. Berg.	Tidskrift for Kemi og Bergvesen
Trans. Acad. Roy. (Stockholm)	Transactions of the Royal Academy (Stockholm)
Trans. Am. Electrochem. Soc.	Transactions of the American Electrochemical Society
Trans. Am. Inst. C. E.	Transactions of the American Institute of Chemical Engineers
Trans. Am. Inst. Min. Eng.	Transactions of the American Institute of Mining Engineering
Trans. Can. Inst. Min.	Transactions of the Canadian Institute of Mining and Metallurgy
Trans. Far. Soc.	Transactions of the Faraday Society
Trans. J. Chem. Soc.	Transactions of the Journal of Chemical Society (London)
Trans. Roy. Soc. (Canada)	Transactions of the Royal Society of Canada
Trans. Roy. Soc. (London)	Transactions of the Royal Society of London
Trans. Roy. Soc. S. Afr.	Transactions of the Royal Society of South Africa
U. S. Pat.	United States Patent
Vid. Sel. Ch. For.	Videnskabs Selskabet Christiania Forhandlinger
Vid. Sel. Ch. Skr.	Videnskabs Selskabet Christiania Skrifter
Wagner's Jahresber.	Wagners Jahresberichte
Wien. Ak. Ber.	Wiener Akademie, Berichte

Z. anal. Chem.	Zeitschrift für analytische Chemie
Z. angew. Chem.	Zeitschrift für angewandte Chemie
Z. anorg. Chem.	Zeitschrift für anorganische u. allgemeine Chemie
Z. electro. angew. phys. Ch.	Zeitschrift für electrochem u. angewandte physikalische Chemie
Z. Chem. Ind. Koll.	Zeitschrift für Chemie und Industrie der Kolloide
Z. f. Chem.	Zeitschrift für Chemie
Z. f. med. Matur.	Zeitschrift für medicine und naturvissenschaften
Z. nahr. Genussm.	Zeitschrift für nahrung und Genussmitteln
Z. physik. Chem.	Zeitschrift für physikalische Chemie
Z. physik. Chem. Unter.	Zeitschrift für physikalischen und chemislchen Unterricht
Z. physiol. Chem.	Zeitschrift für physiologische Chemie (Hoppe Seyler)
Z. unters. Lebensm.	Zeitschrift für untersuchung der Lebensmitte

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Formulas

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c̄ HI, III-2397
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AgCNO c̄ I_2, III-2495
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c̄ $[Co_2(NH_3)_6(OH)_2NO_2]Br_3$, III-685
c̄ $[Co_2(NH_3)_6O_2NH_2OH]Br_3$, III-688
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H_2SiF_6 c̄ $Ir(NH_3)_5Cl(NO_3)_2$, III-2704
H_2TiO_3 c̄ $CoCl_2$, III-97
H_2WO_4 + NaCl c̄ Er_2O_3, III-1551
H_3BO_3 c̄ $(MgO)_3 \cdot Mg(CbO_3)_2$, III-879
$H_3C_6H_5O_7$ c̄ $CuCO_3$, III-979, 984
H_3PO_2 c̄ $Co(OH)_2$, III-240
c̄ $CuSO_4$, III-1279
c̄ $CuCl_2$, III-1081
c̄ I, III-2446
c̄ I_2, III-2549
H_3PO_3 c̄ $CuCl_2$, III-1082
c̄ $CuSO_4$, III-1280
c̄ I_2, III-2550
H_3PO_4 c̄ $CoCO_3$, III-64
c̄ $CoSO_4$, III-265
c̄ $ErCl_3$, III-1511
c̄ Ge, III-1756
H_3PO_4 + Δ c̄ $CuCl_2$, III-1083, 1084
H_3SbO_3 c̄ KIO_3, III-2648
$H_4Fe(CN)_6$ c̄ $Co(NO_3)_2$, III-197
$H_4P_2O_7$ c̄ Er_2O_3, III-1552
H_4SO_5 c̄ H_2O_2, III-2263
Hg c̄ F_2, III-1604
c̄ H_2O_2, III-2264
c̄ I_2S_2, III-2619
c̄ SOClF, III-1656
$HgBr_2$ c̄ Ge, III-1757, 1758
$Hg(CN)_2$ c̄ Cu, III-909
c̄ $CuCr_2O_7$, III-1114
c̄ $Er(SCN)_3$, III-1535
c̄ I_2, III-2551
$Hg(CN)_2$ + H_2O c̄ $H_2S_4O_6$, III-2306
$Hg(CNO)_2$ c̄ Cu, III-910
$Hg(C_2H_5)_2$ c̄ Ga, III-1694
HgCl c̄ $AuCl_3$, III-1978
c̄ Ge, III-1759
$Hg(ClO_4)_2$ c̄ I_2, III-2552
$HgCl_2$ c̄ $[Co(NH_3)_2en_2]Cl_3$, III-539
c̄ $ErCl_3$, III-1512
c̄ Ge, III-1760
c̄ GeS_2, III-1893
c̄ KI, III-2632
$HgCl_2$ + $Hg(CH_3)_2$ c̄ In, III-2328
$HgNO_2$ c̄ C_2H_5I, III-2386
$HgNO_3$ c̄ $K_3Co(CN)_6$, III-320
$Hg(NO_3)_2$ c̄ NH_3, III-2309
HgO c̄ I_2, III-2553
HgO + $KMnO_4$ c̄ $CoCl_2$, III-98
Hg_2O c̄ H_2O_2, III-2264
Hg_2SO_4 c̄ Cu, III-911

I

I c̄ Cu, III-912
c̄ $GeHCl_3$, III-1816
I + Cl c̄ $CoCl_2$, III-99
I^- c̄ F_2, III-1605
c̄ IO_4^-, III-2494
ICl_3, c̄ MF, III-1641
I_2 c̄ $AuCl_3$, III-1979
c̄ Co, III-8, 9
c̄ Cu, III-913
c̄ CuI, III-1124
c̄ F_2, III-1606
c̄ Ga, III-1688
c̄ Ge, III-1761, 1762
c̄ GeI_2, III-1841
c̄ HI, III-2144
c̄ HI_3, III-2144
c̄ H_2S, III-2298
c̄ $K_2Ge_2S_5$, III-1906
c̄ NH_4I, III-2656
c̄ $Na_2H_3IO_6$, III-2670
c̄ RbI, III-2676
I_2 + C_6H_6 c̄ RbI, III-2676
I_2 + H_2O c̄ H_2S, III-2299
I_2 + NaOH c̄ AsH_3, III-2093
I_2 + SO_2 + CH_3OH c̄ $CuCl_2 \cdot 2H_2O$, III-1109
c̄ $CuSO_4 \cdot 5H_2O$, III-1375
In c̄ $InCl_2$, III-2335

K

$K(NH_3)_2Co(NO_2)_4$ c̄ $CoCl_36(NH_3)$, III-146
c̄ $[Co(NH_3)_4(NO_2)_2]Cl$, III-449
c̄ $[Co(NH_3)_4(NO_2)_2]NO_3$, III-572
$K^+ + Cl^-$ c̄ $CoCl_2$, III-100
$K + NH_3$ c̄ Co_2O_3, III-668
$KAuCl_4$ c̄ $[CoSO_3en_2]Cl$, III-436
c̄ $[Co(NH_3)_4(NO_2)_2]NO_3$, III-573
c̄ $[Co_2(NH_3)_8(OH)_2]Cl_4$, III-734
KBr c̄ Au, III-1929
c̄ $AuBr_3$, III-1944
c̄ $AuCl_3$, III-1980
c̄ $[ClCo(C_2H_8N_2)_2OH]Cl$, III-437
c̄ $[CoCO_3en_2]Cl$, III-438
c̄ $[Co(NCS)_2en_2]Cl$, III-439
c̄ $[CoNO_2NCSen_2]Cl$, III-440, 441
c̄ $[ClCo(C_2H_8N_2)_2(C_3H_7N)]Cl_2$, III-514
c̄ $[ClCo(C_2H_8N_2)_2(C_7H_9N)]Cl_2$, III-515
c̄ $[Cl(C_2H_8N_2)_2Co(C_6H_5NH_2)]Cl_2$, III-516
c̄ $[Co(NO_2)_2pn_2]NO_2$, III-560
c̄ $[CoNH_3NO_2en_2](NO_3)_2$, III-591
c̄ $[Co(NH_3)_3Cl(H_2O)_2]SO_4$, III-621
c̄ $Co_2(NO_3)_4(NO)_2(NH_3)_{10}\cdot 2H_2O$, III-655
c̄ $[Co_2NHHNO_3O_2](NO_3)_3$, III-764
c̄ $Cu(OH)_2$, III-1212
c̄ $[Ir(NO_2)_2(NH_3)_4]Cl$, III-2745
c̄ $KH(IO_3)_2$, III-2620
KBrO c̄ $CuSO_4$, III-1281
KCN c̄ Au, III-1930, 1931, 1932, 1933
c̄ AuCN, III-2033
c̄ $AuCl_3$, III-1981
c̄ $Au_2O_3\cdot 4NH_3$, III-2033
c̄ Au_2S_3, III-2034
c̄ C_2H_5I, III-2392
c̄ Co, III-10
c̄ $Co(CN)_2$, III-45, 101
c̄ $Co(C_2O_2H_3)_2$, III-65
c̄ $(CoCl_2)$, III-101
c̄ $CoCl_2$, III-102, 103
c̄ $CoSO_4$, III-266
c̄ $Cu(NH_3)_4Cl_2$, III-1393
c̄ $CuSO_4$, III-1282, 1283
c̄ $CuSO_4\cdot 4NH_3$, III-1381, 1382
c̄ $Cu_2(CN)_2$, III-1415
c̄ ICl, III-2468
c̄ I_2, III-2554
c̄ $K_2[IrCl_6]$, III-2753
KCN + C c̄ GeO_2, III-1878
KCN + H_2O c̄ Cu_2Se, III-1475
KCN + O_2 c̄ Au, III-1934
KCNO c̄ $CoCl_2$, III-104
c̄ ICl, III-2469
KCNS c̄ $[CoBr_2en_2]Br$, III-355, 356
c̄ $[CoNCSBren_2]Br$, III-605
c̄ $[CoSO_2(CH_2COO)_2en_2]Br\cdot 2H_2O$, III-368
c̄ $[CoCO_3en_2]Cl$, III-442
c̄ $CoCl_2$, III-105
c̄ $[CoNO_2NCSen_2]NO_2$, III-562
c̄ $[CoClNO_2en_2]NO_3$, III-574
c̄ $[Co(NH_3)_3C_2O_4H_2O]NO_3$, III-575
c̄ $[Co(NO_2)_2en_2]NO_3$, III-576, 577
c̄ $[CoNH_3NO_2en_2](NO_3)_2$, III-593, 594
c̄ $[CoH_2ONCSen_2]S_2O_6$, III-628
c̄ $CoSO_4$, III-267
c̄ ICl, III-2470
KCNS + $(NH_2(CH_3)C_6H_3)_2$ c̄ $CuSO_4$, III-1286
$KC_2H_3O_2$ c̄ $KH_2Co_3(CN)_{11}\cdot H_2O$, III-819
$KC_{16}H_{31}O_2$ c̄ $CuSO_4$, III-1288
KCl c̄ CuCl, III-1044
c̄ H_2SiF_6, III-1626
c̄ ICl, III-2471
c̄ $Na_2OI_2O_7\cdot 12WO_3$, III-2671
c̄ Na_2SiF_6, III-1655
c̄ $Na_3Co(NO_2)_6$, III-328, 330
c̄ $UO_2(IO_3)_2$, III-2681
KCl + $BaCl_2$ + $NaNO_2$ c̄ $CuCl_2$, III-1085
KCl + HCl c̄ Ir, III-2701
KCl + H_2F_2 c̄ GeO, III-1853
$KClO_3$ c̄ HCl, III-2117
$KClO_3$ + H_2SO_4 c̄ Cu, III-914
KF c̄ GeF_4, III-1810
c̄ $HCbO_3$, III-868
KF + HF c̄ $CbOF_3\cdot 2KF$, III-840
$(KF)_3HF\cdot PbF_4$ c̄ Ir, III-2688
$KHCO_3$ c̄ $Co(NO_3)_2$, III-198, 199
$KHCO_3$ + H_2O_2 c̄ $CoCO_3$, III-66
KHF_2 c̄ $CoCl_2$, III-106
c̄ D_2O, III-2083
c̄ GeF_4, III-1811
KHS c̄ $CoCl_2$, III-107
$KHSO_4$ c̄ $Au(OH)SO_4$, III-2022
c̄ Er_2O_3, III-1553
c̄ $Fe(CbO_3)_2$, III-866
$KH_2CO_3(CN)_{11}\cdot H_2O$ c̄ $CuSO_4$, III-1289
KH_2PO_4 c̄ D_2O, III-2083
KI c̄ AuCOCl, III-1950
c̄ $AuCl_3$, III-1982, 1983
c̄ $[CoBr_2en_2]Br$, III-354
c̄ $[SO_2(CH_2COO)_2Co(NH_2C_2H_4NH_2)_2]Br\cdot 2H_2O$, III-365

c̄ $CoBr_2C_6H_7O_2 \cdot en_2 \cdot H_2O$, III-31
c̄ $[CoH_2O(OH)en_2]Br_2$, III-379
c̄ $[CoNH_3NO_2en_2]Br_2$, III-380
c̄ $[CoNH_3H_2Oen_2]Br_3$, III-404
c̄ $[CoC_2O_4en_2]Cl$, III-443
c̄ $[CoCO_3en_2]Cl$, III-444
c̄ $[Co(NCS)_2en_2]Cl$, III-445
c̄ $[Co(NH_3)_2Cl_2en]Cl$, III-446
c̄ $[CoNO_2NCSen_2]Cl$, III-447, 448
c̄ $[Co(C_2H_8N_2)_2Cl(C_6H_5NH_2)]Cl_2$, III-517
c̄ $[CoCl(C_2H_8N_2)_2(C_7H_7N)]Cl_2$, III-518
c̄ $[CoCl(C_2H_8N_2)_2(C_7H_9N)]Cl_2$, III-519
c̄ $[Co(C_3H_6O_2N)(C_2H_8N_2)_2]Cl_2$, III-520
c̄ $[Copn_3]Cl_3$, III-540
c̄ $[Co(NH_3)_2en_2]Cl_3$, III-541
c̄ $[Co(NH_3)_2en_2]Cl_4, C_2O_4$, III-545
c̄ $[Co(NO_2)_2pn_2]NO_2$, III-561
c̄ $Co(NO_3)_2$, III-200
c̄ $[CoNH_3NO_2en_2](NO_3)_2$, III-592
c̄ $[Co(NH_3)_2en_2](NO_3)_3$, III-601
c̄ $Co(OH)_3$, III-282
c̄ $[Co_2(NH_3)_6(OH)_2C_2H_3O_2]Br_3$, III-694
c̄ $[Co_2(NH_3)_8(OH)_2]Cl_4$, III-735
c̄ $[Co_2(NH_3)_6(H_2O)_2NH_2OH](NO_3)_4$, III-784
c̄ $[Co_2(NH_3)_8NH_2NO_2](NO_3)_4$, III-785
c̄ $Cu(C_2H_3O_2)_2$, III-902, 996
c̄ $Cu(NO_3)_2$, III-1146
c̄ $CuSO_4$, III-1277, 1290, 1291, 1292
c̄ $CuSO_4 \cdot 4NH_3 \cdot H_2O$, III-1384
c̄ H_2O_2, III-2265
c̄ ICl, III-2472
c̄ I_2, III-2555, 2556, 2557, 2558
c̄ $Ir(NH_3)_5Cl(NO_3)_2$, III-2705
c̄ $[IrCl_2(NH_3)_4]Cl$, III-2746
c̄ $[Ir(NO_2)_2(NH_3)_4]Cl$, III-2747
c̄ $[Ir(NH_3)_5NO_2]Cl_2$, III-2750
c̄ KIO_3, III-2649, 2650
c̄ KIO_4, III-2652
c̄ NO_2F, III-1644, 1645
KI + $HC_2H_3O_2$ + $Na_2S_2O_3$ c̄ $[Co_2NH_2O_2en_4](NO_3)_3$, III-765
KI + HCl c̄ $Co(OH)_3$, III-246
KI + HNO_3 c̄ $Co_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O$, III-656
KI + H_2SO_4 c̄ $Co(OH)_3$, III-247
KI + H_3BO_3 c̄ KIO_4, III-2653
KI + $NH_2C_6H_4C_6H_4NH_2$ c̄ $CuSO_4$, III-1293
KIO_3 c̄ $Co(NO_3)_2$, III-201
c̄ $Co(OH)_2$, III-241
c̄ $CoSO_4$, III-268
c̄ $InCl_3$, III-2338
c̄ KI, III-2633
KIO_3 + HCl c̄ CuSCN, III-1239
KIO_3 + H_2SiF_6 c̄ KI, III-2634
$KMnO_4$ c̄ $AuCl_3$, III-1984
c̄ CuOH, III-1203
c̄ HCl, III-2118
c̄ H_2O_2, III-2266
c̄ KI, III-2636
$KMnO_4$ + H_2O_2 c̄ $CoSO_4$, III-269
$KMnO_4$ + H_2SO_4 c̄ CuCNS, III-970
c̄ GeH, III-1813
c̄ H_2O_2, III-2267
$KMnO_4$ + HgO c̄ $CoCl_2$, III-108
KNH_2 c̄ $Co(SCN)_2$, III-297
c̄ $CuNH_2$, III-1132
c̄ $Cu(NO_3)_2$, III-1147
c̄ $Cu(NO_3)_2 \cdot 4NH_3$, III-1157
c̄ SnI_4, III-2679
KNO_2 c̄ Co^{++}, III-18
c̄ $CoCl_2$, III-109
c̄ $CoCl_3$, III-137
c̄ $IrCl_3$, III-2696
c̄ $IrCl_4$, III-2696
c̄ K_2IrCl_6, III-2725
c̄ K_3IrCl_6, III-2725
c̄ $Na_2(IrCl_6)$, III-2734
KNO_2 + HNO_2 c̄ $CoCO_3$, III-67
c̄ $CoCl_2$, III-110
KNO_2 + NH_3 c̄ $CoSO_4$, III-270
KNO_2 c̄ $[CoC_3H_2O_4en_2]C_3H_3O_4$, III-503
c̄ $[Co(NCS)_2en_2]Cl$, III-450
c̄ $[CoNO_2NCSen_2]Cl$, III-451
c̄ HCl, III-2119
c̄ KF, III-1632
KNO_2 + e c̄ $CuSO_4$, III-1294
KOBr c̄ KI, III-2637
KOH c̄ AgF, III-1581
c̄ $Au(OH)_2NH_2$, III-2023
c̄ Au_2O_3, III-2031
c̄ CNI, III-2384
c̄ C_7H_7OCOOH, III-1372
c̄ Cb_2O_5, III-854
c̄ CoC_2O_4, III-76
c̄ $[Co(NCS)_2en_2]Cl$, III-452
c̄ $[Co(NH_3)_3(C_2O_4)_2]H$, III-338
c̄ $CoNH_4(SO_3)_2 \cdot 4NH_3$, III-163
c̄ $Co(OH)_3$, III-242
c̄ $[CoClNCSen_2]SCN$, III-613
c̄ $Co(SO_4)$, III-271, 272
c̄ $CuCl_2$, III-1086

c̄ $CuSO_4$, III-1295
c̄ $Ga(C_2H_5)_2OH$, III-1707
c̄ $Ga(C_2H_5)_3 \cdot NH_3$, III-1707
c̄ $GeBr_4$, III-1776
c̄ $GeCl_2$, III-1787
c̄ GeO_2, III-1879
c̄ $HAuCl_4$, III-2045
c̄ $HI \cdot HCN$, III-2417
c̄ ICN, III-2454
c̄ ICl, III-2473, 2474
c̄ I_2, III-2559, 2560, 2561
c̄ I_2O_4, III-2604, 2605
c̄ In^{+++}, III-2330
c̄ $InCl_3$, III-2339
c̄ $Ir(NH_3)_5Cl_3$, III-2714
c̄ $KAuBr_2$, III-2051
c̄ $KIr(H_2O)_2(C_2O_4)_2$, III-2722
c̄ K_2SiF_6, III-1639
c̄ $K_{10}Cu_3(C_6H_5O_7)_4(CO_3)_2$, III-1478
c̄ NO_2F, III-1646
c̄ Na_2IrCl_6, III-2735
c̄ SnI_4, III-2680
KOH + CO c̄ $CoCl_2$, III-111
KOH + H_2O_2 c̄ Cb_2O_5, III-855
KOH + $K_3C_6H_5O_7$ + $H_3C_6H_5O_7$ c̄ $CuSO_4$, III-1296
KOH + N_2O_3 c̄ $Co_2(CO_3)_3$, III-634
KOH + NaCl c̄ Na_2IrCl_6, III-2735
KOH + O_2 c̄ $CoBr_2 \cdot 6H_2O$, III-22
KOI c̄ KCN, III-2384
KSCN c̄ $[CoH_2ONCSen_2]Br_2$, III-381
c̄ $[CoH_2O(OH)en_2]Br_2$, III-382
c̄ $[Co(H_2O)_2en_2]Br_3$, III-405
c̄ $[CoClNO_2en_2]Cl$, III-453
c̄ $[Co(NH_3)_2Cl_2en]Cl$, III-454
c̄ $[Co(NCS)_2en_2]Cl$, III-455
c̄ $[CoNO_2NCSen_2]Cl$, III-456
c̄ $[CoSO_3en_2]Cl$, III-457
c̄ $[CoCl_2en_2]Cl$, III-458
c̄ $[CoHN_3Clen_2]Cl_2$, III-521, 522
c̄ $[Co(NH_3)_2en_2]Cl_3$, III-542
c̄ $[Copn_3]I_3$, III-554
c̄ $[Co_2(NH_3)_6(OH)_2C_2H_2O_2]Br_3$, III-695
c̄ $[Co_2(OH)_2en_4]Br_4$, III-700
c̄ $[CoH_2ONCSen_2]_2S_2O_6$, III-811
c̄ CuCN, III-960
c̄ $CuCl_2$, III-1078
c̄ $CuSO_4$, III-1284, 1287
KSCN + C_5H_5N c̄ $CuSO_4$, III-1285
KSCN + HNO_3 + NH_4Br c̄ $[Co_2NH_2O_2en_4](NO_3)_3$, III-766
KSCN + $TiCl_2$ c̄ $CuCl_2$, III-1087
$KSCSN_3$ c̄ I_2, III-2562
$(KSbOC_4H_4O_6)_2H_2O$ c̄ HCl, III-2120
K_2CO_3 c̄ $CbOF_3 \cdot 2KF$, III-841
c̄ Cb_2O_5, III-856, 857, 859
c̄ $[Co(NH_3)_2Cl_2en]Cl$, III-459
c̄ $CoCl_2$, III-112
c̄ $CuCO_3$, III-981
c̄ $Cu(NO_3)_2$, III-901
c̄ $CuSO_4$, III-1297
c̄ $K_3Cb_6O_{19}$, III-878
K_2CO_3 + $H_2C_2O_4$ c̄ Cb_2O_5, III-858
K_2CO_3 + $K_3C_6H_5O_7$ c̄ $Cu_3(C_6H_5O_7)_2$, III-1478
$K_2C_2O_4$ c̄ CoC_2O_4, III-77
c̄ $Cu_2Fe(CN)_6$, III-1423
c̄ I_2, III-2563
c̄ $In_2(SO_4)_3$, III-2372
c̄ K_2IrCl_6, III-2724
c̄ K_3IrCl_6, III-2728, 2729
$K_2C_3H_2O_4$ c̄ $CoC_3H_2O_4$, III-79
K_2CrO_4 c̄ $CoCl_3 \cdot H_2O \cdot 5NH_3$, III-142
c̄ $Co(NO_3)_2$, III-202
c̄ $Co(NO_3)_3 \cdot (H_2O)_2 \cdot 4NH_3$, III-216
c̄ $CO_2(NO_3)_4(NO)_2(NH_3)_{10} \cdot H_2O$, III-657
c̄ $Cu(NO_3)_2$, III-1148
c̄ HCl, III-2121
c̄ $InCl_3$, III-2340
c̄ KI, III-2638
$K_2Cr_2O_7$ c̄ $[Co(NH_3)_4(NO_2)_2]NO_3$, III-578
c̄ HCl, III-2121
c̄ H_2O_2, III-2268
c̄ KI, III-2639
$K_2Cr_2O_7$ + H_2SO_4 c̄ Cu_2O, III-1438
K_2F_2 c̄ Cb_2O_5, III-853
c̄ H_2GeF_6, III-1901
$K_2HC_6H_5O_7$ c̄ HCl, III-2122
$K_2Hg(CNS)_4$ c̄ $CuCl_2$, III-1088
K_2HgI_4 c̄ $Co(NO_3)_2 \cdot 6NH_3$, III-213
c̄ $Cu(NO_3)_2 \cdot 4(NH_3)$, III-1158
K_2O_4 c̄ Au, III-1935
$K_2Pb(NO_2)_4$ c̄ $K_2Co(NO_2)_4$, III-308
K_2PtCl_4 c̄ $[CoNH_3Bren_2]Br_2$, III-383
c̄ $[Co(NH_3)H_2Oen_2]Br_3$, III-406
c̄ $Co(NO_2)Cl_2(H_2O) \cdot 4NH_2$, III-169
c̄ $[Co(NH_3)_4(NO_2)_2]NO_3$, III-579
c̄ $[Co_2(NH_3)_6AcNH_2OH]Cl_3$, III-755
c̄ $[Co_2(NH_3)_8(OH)_2]Cl_4$, III-736
K_2PtCl_6 c̄ $[CoSO_3en_2]Cl$, III-460
K_2S c̄ $CuNO_3$, III-1138
K_2SO_4 c̄ $Cu(CH_3COO)_2$, III-954

c̄ $Er_2(SO_4)_3$, III-1553, 1562, 1563
c̄ $Gd_2(SO_4)_3$, III-1684
c̄ $[Ir(NO_2)_2(NH_3)_4]Cl$, III-2748
c̄ $Ir_2(SO_4)_3$, III-2721
$K_2SO_4 + H_2O$ c̄ $CuSO_4$, III-1298
$K_2S_2O_3$ c̄ $CuSO_4$, III-1346
$K_2S_2O_8$ c̄ $Co(OH)_2$, III-272
c̄ H_2O_2, III-2269
K_2SeO_4 c̄ $CoSO_4$, III-273
c̄ $Er_2(SeO_4)_3$, III-1570
c̄ $Gd_2(SeO_4)_3$, III-1687
K_2TeO_4 c̄ $Cu(NO_3)_2$, III-1149
$K_3C_6H_5O_7 \cdot H_2O$ c̄ $Cu_3(C_6H_5O_7)_2 \cdot CuO \cdot 2H_2O$, III-1479
c̄ HCl, III-2122
$K_3[Co(CN)_6]$ c̄ $[Copn_3]I_3$, III-555
c̄ $Gd(NO_3)_3$, III-1666
$K_3[Cr(CN)_6]$ c̄ $[Coen_3]Cl_3$, III-543
c̄ $[Copn_3]I_3$, III-556
c̄ $Co(NO_3)_2$, III-203
c̄ CuCl, III-1045
c̄ $CuSO_4$, III-1299
$K_3[Cr(C_2O_4)_3]$ c̄ $[Co(NH_3)_6(NO_3)_3]$, III-337
c̄ $[Coen_3]Br_3$, III-407
c̄ $[Copn_3]I_3$, III-557
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$K_4Fe(CN)_6$ c̄ $CoSO_4$, III-274
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c̄ $Co_2Fe(CN)_6$, III-637
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$LiNO_3$ c̄ $[CoH_2OClen_2]Br_2$, III-384
c̄ $[CoNH_3Bren_2]Br_2$, III-385
c̄ $[CoBrNCSen_2]ClO_4$, III-495
c̄ $[CoClNCSen_2]ClO_4$, III-494
c̄ $[CoClSCNen_2]ClO_4$, III-627
LiOH c̄ CuCN, III-965
$Li_2C_2O_4$ c̄ Li_2IrCl_6, III-2731
Li_2SO_4 c̄ $CuSO_4$, III-1303
$Li_4Fe(CN)_6$ c̄ CuCN, III-962
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c̄ D_2O, III-2084
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$MnCl_4$ c̄ Au, III-1936
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c̄ HI, III-2411
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MnO_4 c̄ KI, III-2635
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c̄ $NaIO_3$, III-2664
MoO_3 + HF c̄ CuO, III-1185

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$NH_2OH \cdot H_2SO_4$ c̄ $Cu(OH)_2$, III-1213
$(NH_2OH)_2 \cdot HCl + KOH$ c̄ $CuSO_4 \cdot 5H_2O$, III-1376
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c̄ $[CoNH_3Cl_2H_2Oen]Cl$, III-464
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c̄ $[CoCl_2en_2]Cl$, III-467
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$NH_3 + HBr$ c̄ $[Co_2(NH_3)_6Cl_3O_2OH]Cl$, III-704
$NH_3 + HCOOH$ c̄ $Cu(OH)_2$, III-1214
$NH_3 + H_2O$ c̄ $Cu(CN)_2$, III-1283
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c̄ SO_2, III-2300
$NH_3 + KI$ c̄ $[CoC_2O_4en_2]Cl$, III-465
$NH_3 + KSCN$ c̄ $[CoClNCSen_2]SCN$, III-614
c̄ $[CoClNCSen_2](SCN)_2$, III-616
$NH_3 + (NH_4)_2SO_4$ c̄ $Cu(OH)_2$, III-1215
$NH_3 + (NH_4)_4Fe(CN)_6$ c̄ $CuCl_2$, III-1090
$NH_3 + NaBr$ c̄ $[CoCl_2en_2]Cl$, III-463
$NH_3 + \Delta$ c̄ $Ga(CH_3)_2$, III-1698
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c̄ $[Co_2(NH_3)_6O_2Cl_2NH_2]Cl_2$, III-716

c̄ $[Co_2(NH_3)_6NH_2OHO_2]Cl_3$, III-788
c̄ $[Co_2(NH_3)_6H_2ONH_2OH](NO_3)_3$, III-770
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c̄ Cu, III-931
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c̄ CuO, III-1194, 1195
c̄ $CuSO_4$, III-1195
c̄ Ge, III-1768, 1769
c̄ HI, III-2414
c̄ HIO_3, III-2418
c̄ I_2, III-2594, 2595, 2596
$SO_2 + H_2O$ c̄ $Er(OH)_3$, III-1533
$SO_2 + NH_4OH$ c̄ $CuSO_4$, III-1360
$SO_2 + O_2$ c̄ CuO, III-1196
$SO_2(CH_2COOH)_2$ c̄ $[CoCO_3en_2]Br$, III-365
SO_2Cl_2 c̄ Au, III-1940
$SO_2Cl_2 + SbCl_5$ c̄ SbF_3, III-1658
SO_3 c̄ H_2F_2, III-1622
c̄ I_2O_4, III-2606
c̄ I_2O_5, III-2616
S_2 c̄ Si, III-1476
S_2Br_2 c̄ ICl, III-2482
S_2Cl_2 c̄ Cb_2O_5, III-863
c̄ $(CoNi)_2S_4$, III-663
c̄ $CuFeS_2$, III-1116
$S_2O_8^{--}$ c̄ Cu, III-932
$SbCl_2F$ c̄ $GeCl_4$, III-1804
SbF_3 c̄ $GeCl_4$, III-1804
Se c̄ Cb, III-826
c̄ CuCN, III-968
c̄ F_2, III-1611
c̄ Ge, III-1770
$SeCl_4$ c̄ Cu, III-933
$SeO_2 + H_2O$ c̄ $CoSeO_3$, III-299
c̄ $CuSeO_3$, III-1389
c̄ $Er_2(SeO_3)_3$, III-1569
c̄ $[In_2(SeO_3)_3]_3 \cdot [In(OH)_3]_2 \cdot 22H_2O$, III-2374
Se_2Cl_2 c̄ Cu, III-933
c̄ Cu_2Se, III-933
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$SnCl_2$ c̄ $CuSO_4$, III-1361
$SnCl_2 + HCl$ c̄ $Co(OH)_3$, III-248
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$SnCl_4$ c̄ $CoCl_2 \cdot 6H_2O$, III-134
c̄ $Cu(C_{15}H_{33}O_2)_2$, III-1027
Sn_4P_3 c̄ HCl, III-2133
$Sr(CN)_2$ c̄ CuCN, III-956
$SrCl_2$ c̄ D_2O, III-2083
$Sr(HC_4H_4O_6)_2$ c̄ $CoSO_4$, III-294
$Sr(NO_3)_2$ c̄ $Na_4I_2O_9$, III-2673
SrS_2O_3 c̄ $CoSO_4$, III-295
$Sr_2Fe(CN)_6$ c̄ $Cu_2Fe(CN)_6$, III-1425

T

Te c̄ Au, III-1941
c̄ Co, III-15
c̄ Cu, III-937
$TiCl_3$ c̄ $CuCl_2$, III-1078
$TiCl_3 + HCl$ c̄ H_2O_2, III-2276
$Ti_2(SO_4)_3$ c̄ $Co(OH)_3$, III-249
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Tl_2CO_3 c̄ $Na_3Co(NO_2)_6$, III-331
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Tl_2SO_4 c̄ $Co(NH_3)_6(NO_2)_6Co$, III-640

W

WCl_6 c̄ AsF_3, III-1583

Z

Zn c̄ $AuBr_3$, III-1945
c̄ $AuCl_3$, III-2004

c̄ CH_3I, III-2381
c̄ $C_2O_4Br_2$, III-2063
c̄ C_3H_5I, III-2394
c̄ Cu, III-938
c̄ $CuCl_2$, III-1102
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c̄ Cu_2Si, III-1477
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c̄ $InCl_3$, III-2350
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$Zn(C_2H_5)_2$ c̄ $GeCl_4$, III-1805
$ZnCl_2$ c̄ $KCbO_3$, III-873
ZnS c̄ $Cu(NO_3)_2$, III-1154
$ZnSO_4$ c̄ $K_3Co(CN)_6$, III-321

β and γ Rays

β and γ Rays c̄ H_2O, III-2243

Decomposition

Decomp. c̄ AuCoCl, III-1952, 1953
c̄ $[Co(NH_3)_3C_2O_4H_2O]Cl$, III-489
c̄ $CoCl_3 \cdot H_2O \cdot 5NH_3$, III-141
c̄ $[CoNH_3Clen_2](NO_2)_2$, III-566
c̄ $[Co_2(NH_3)_8BrH_2ONH_2]Br_4$, III-703
c̄ $[Co_2(NH_3)_8H_2ONH_2Cl]Cl_4$, III-749
c̄ CuI_2, III-1129
c̄ D_2O_2, III-2089
c̄ GeH_3Cl, III-1829
c̄ GeI_2, III-1843
c̄ $Ge(OH)_2$, III-1857
c̄ H_2O_2, III-2291, 2292, 2293
c̄ ICl_3, III-2488
c̄ $I(OH)_3$, III-2490

ε Electric Current

ε c̄ $AuCl_3$, III-2016, 2017
c̄ CH_3F, III-1585, 1586
c̄ $Co(CH_3COO)_2$, III-39
c̄ $CoCl_2$, III-132
c̄ Cu^{++}, III-943
c̄ $Cu(H_2O)x^{++}$, III-1119
c̄ $Cu(NO_2)_2$, III-1136
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c̄ $CuSO_4$, III-1363, 1364, 1365
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c̄ F_2, III-1613
c̄ $Ga_2(SO_4)_3$, III-1721
c̄ GeO_2, III-1884, 1885
c̄ HCl, III-2134, 2135
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c̄ KHF_2, III-1635, 1636
c̄ KI, III-2644, 2645
c̄ Na_3GaO_3, III-1725
ε + (NaCl) c̄ NH_3, III-2310

(Δ) Heat

Δ c̄ $AuCl_3$, III-2018
c̄ $(CH_3CH_2CH_2)_2AuCN$, III-2036
c̄ $(CH_3)_3NHAuCl_4$, III-2037
c̄ $C_6H_5N_2F$, III-1587
c̄ $[CoBr_2en_2]Br$, III-366, 367
c̄ $[CoH_2OClen_2]Br_2$, III-392, 393, 394, 495
c̄ $CoBr_2 \cdot 6H_2O$, III-24
c̄ $[Co(H_2O)_2en_2]Br_3$, III-410
c̄ $Co(CH_3COO)_3$, III-44
c̄ $Co(CO)_4$, III-75
c̄ CoC_2O_4, III-78
c̄ $Co(C_5H_5N)_4SiF_6$, III-80
c̄ $[CoCl_2en_2]Cl$, III-492
c̄ $[Co(NH_3)_5(NO_2)]Cl_2$, III-536
c̄ $CoCl_2 \cdot 6NH_3$, III-148
c̄ $CoHPO_3$, III-154
c̄ $Co(H_2PO_2)_2$, III-155
c̄ $CoI_2 \cdot 6H_2O$, III-158
c̄ $Co(NH_3)_3Cl_3$, III-162, 195
c̄ $[Co(NO_2)_2en_2]NO_2$, III-563
c̄ $CoSO_4$, III-296
c̄ $[CoH_2OOHen_2]S_2O_6$, III-632
c̄ $Co_2(NO_2)_6(KNO_2)_6 \cdot 3H_2O$, III-647
c̄ $2CoCO_3 \cdot Co(OH)_2$, III-813
c̄ $CuBr_2$, III-951
c̄ $Cu(CH_3COO)_2 \cdot H_2O$, III-955
c̄ $Cu(CN)_2$, III-972
c̄ $Cu(C_2H_3O_2)_2$, III-1004
c̄ $Cu(C_2H_3O_2)_2 \cdot C_5H_5N$, III-1010
c̄ $CuCl_2$, III-1103, 1104, 1105

c̄ $CuCl_2 \cdot CuO$, III-1108
c̄ $CuCl_2 \cdot (KCl)_2 \cdot 2H_2O$, III-1112
c̄ $Cu(HCO_2)_2$, III-1117
c̄ $Cu(H_2NSO_4)_2$, III-1118
c̄ $Cu(IO_3)_2 \cdot (NH_3)_4 \cdot 2H_2O$, III-1128
c̄ CuI_2, III-1130
c̄ $Cu(NO_2)_2$, III-1137
c̄ $Cu(NO_3)_2$, III-1155
c̄ CuO, III-1198, 1199
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c̄ $Cu(SCSOC_2H_5)_2$, III-1243
c̄ $CuSO_4$, III-1366
c̄ $CuSiF_6 \cdot 6H_2O$, III-1392
c̄ $Cu_2(CH_3COO)_2$, III-955
c̄ $Cu_2ClH_3O_3$, III-1399
c̄ $Cu_2ClO_3H_3$, III-1400
c̄ Cu_2HIO_6, III-1427
c̄ Cu_2O, III-1444
c̄ $(Cu_2P_2O_7)_2 \cdot Na_4P_2O_7$, III-1448
c̄ $Cu_2S_4O_6$, III-1473
c̄ Cu_2S_6, III-1474
c̄ Cu_3PS_4, III-1481
c̄ $Cu_4H_6O_{10}S$, III-1482
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c̄ $Er(COOH)_3$, III-1507
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c̄ $In(NO_3)_3$, III-2351
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c̄ Ir_2O_3, III-2720
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c̄ KHF_2, III-1637
c̄ NH_4CoPO_4, III-327
c̄ $NaGeH_3$, III-1909
c̄ SO_3HF, III-1657
Δ + O_2 c̄ H_2, III-2215
+ KSCN c̄ $[CoCl_2en_2]Cl$, III-490, 491
+ NH_3 c̄ $Cu(IO_3)_2 \cdot 4NH_3$, III-1128
+ N_2 c̄ $NaHF_2$, III-1653

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Light c̄ $AuCl_3$, III-2012
c̄ H_2O_2, III-2283
c̄ $K_2Cu(C_2O_4)_2$, III-1488
c̄ $K_3[Co(CN)_6]$, III-322
c̄ $K_3Co(C_2O_4)_3$, III-324
Light + Cl_2 c̄ H_2, III-2217

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c̄ $Cu_2Cl_2(CO)_2 \cdot 4H_2O$, III-1412

Index to Reagents

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Adenine c̄ $AuCl_3$, III-2005
Air c̄ $CuBrC_2H_3O_2 \cdot 5NH_3$, III-949
d-1 Alanine c̄ $CuCO_3$, III-987
Aldoses + NaOH c̄ I_2, III-2582, 2598
3 Amino-4-bromo-benzoic acid c̄ $Cu(C_2H_3O_2)_2$, III-1005
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Betaine c̄ $AuCl_3$, III-2006
Blood catalase c̄ H_2O_2, III-2277
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Calcium-3-bromo-2 toluene sulfonate c̄ $CuSO_4$, III-1369
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Oleic acid c̄ H_2O_2, III-2287

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c̄ H_2O_2, III-2288
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c̄ $CuSO_4$, III-1374

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Stearic acid c̄ H_2, III-2223
c̄ H_2O_2, III-2289
Succinic acid c̄ $[CoCl_2en_2]Cl$, III-487

Trimethyl putrescine c̄ $AuCl_3$, III-2015
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In order to facilitate the location of any desired products obtained in the foregoing reactions the following index has been included. The names of compounds follow the list of formulas.

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